VON KARMAN CENTER

CHEMICAL PRODUCTS DIVISION

RESEARCH ON PROCESSES FOR UTILIZATION OF LUNAR RESOURCES

A REPORT TO

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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AEROJET GENERAL TIRE GENERAL

RESEARCH ON PROCESSES FOR UTILIZATION OF LUNAR RESOURCES

a report to

OFFICE OF ADVANCED RESEARCH AND TECHNOLOGY
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AEROJET-GENERAL CORPORATION
A SUBSIDIARY OF THE GENERAL TIRE & RUBBER COMPANY

This report is submitted in partial fulfillment of Contract NAS 7-225. The period covered by the report is 16 November 1963 through 15 July 1964.

AEROJET-GENERAL CORPORATION

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ABSTRACT

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Laboratory apparatus for studying the reduction of igneous rock with carbon, hydrogen, and methane was designed, fabricated, and operated. Several experiments demonstrated that with the use of specially designed inlet tubes, methane reacts with silicate materials at 1600°C to form carbon monoxide, hydrogen, silicon, carbon, and slag. Quantitative recovery of carbon was achieved. Reactor materials were found which allow the reaction to run at 1600°C for long time periods.

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I. OBJECTIVE

The objective of this program was to study the reduction of natural silicates with methane, carbon, hydrogen, and mixtures of methane and hydrogen. Sufficient data were obtained to permit a preliminary evaluation of this reaction as a step in the reduction of silicate materials (lunar raw materials) to produce oxygen.

II. SUMMARY, CONCLUSIONS, AND RECOMMENDATIONS

A. SUMMARY

1. Task 1, Design, Fabrication, and Testing

Task 1 was devoted to the design of bench-scale equipment for use in determining the feasibility of the first step (the reduction of silicates with methane) in the Aerojet Carbothermal Process for the manufacture of oxygen from lunar minerals. This equipment consisted of an induction-heated rock reactor (50 cu cm capacity) together with its auxiliary flow system. Modifications in the design, especially of the gas inlet tubes, occurred throughout the program. Schematic flow diagrams of the apparatus are shown in Figures 1 and 2.

Standard industrial and laboratory equipment and materials were used wherever possible in the fabrication of the unit in order to speed and simplify construction. The fabricated unit and its components are shown in Figures 3, 4, and 5.

Ten runs were devoted to the testing of the apparatus. Temperatures as high as 1900°C were reached while excellent heat control was maintained. Table 1 summarizes the ten test runs.

2. Task 2, Reduction of Igneous Rock

Data obtained from a total of 22 runs indicate that the first step of the carbothermal process can be adapted to lunar silicates. The significant achievements attained are summarized below.

a. Reduction of Silicates

Reduction of silicates with carbon or with methane produced carbon monoxide and significant amounts of silicon. Near-quantitative recoveries of carbon were achieved. It is most significant that these results were obtained with all of the natural silicates used (i.e., tektites, basalt, and granite).

b. Inlet Tube Design

An inlet tube design which was evolved during the program allowed the delivery of methane into molten rock for extended time periods without the loss of carbon or the premature cracking of methane in the inlet tube. This design, represented in Figure 6, consists of a triple walled inlet tube terminating in a "bell" which extends into the molten rock. Cooling hydrogen can circulate in the outer jacket to cool the rapidly flowing methane which enters the "bell" via the center tube. The "bell" provides a lower linear velocity for the methane, permitting methane cracking to occur above the melt surface. No methane escaped through the melt when this type of inlet was used, and quantitative recoveries of carbon (as either CO or elemental carbon) were achieved.

c. Reaction Temperature

With the use of zirconia crucibles and inlet tubes fabricated from zirconia, alumina, and Zircoa Cast, it was determined that the optimum temperature for the silicate reduction was near 1600°C. Below this temperature, carbon monoxide production was low due to formation of carbides; above this temperature, carbon reacted with the ceramic materials of the inlet tube and crucible.

B. CONCLUSIONS

1. The objectives of the program have been achieved. The feasibility of the quantitative recovery of carbon when methane is reacted with molten silicates was demonstrated. Most of the carbon was obtained as carbon monoxide; the remainder was obtained as elemental carbon in usable form. It was demonstrated, within experimental error, that the elemental carbon can be quantitatively converted to carbon monoxide by reaction with natural silicates. The silicates are reduced in this reaction to form various metals, including silicon.

2. Reaction conditions and new inlet tube designs were found which allowed the reaction to run for periods longer than 8 hours at 1600°C without mechanical failure or chemical attack of the reactor materials.

3. It was demonstrated that if the silicate material contains water in any form, this water is obtained as a by-product in this process.

4. Numerous difficulties were experienced with induction heating of the reactor, causing many runs to be terminated prematurely. Resistance heating should be used in future furnace designs.

C. RECOMMENDATIONS

The Aerojet Carbothermal Process has three essential steps: (1) the reduction of silicate with methane to form carbon monoxide and hydrogen; (2) the reduction of carbon monoxide with hydrogen to form methane and water; and (3) the electrolysis of water to form hydrogen and oxygen. The process is cyclic in nature, with the methane and hydrogen returned to the system. The feasibility of Step 2 was demonstrated on the initial increment of Contract NAS 7-225 (Reference 1).

1. The study of Steps 1 and 2 of the process should be continued on a larger scale to provide more precise mass balance data.

2. Experiments of longer duration should be performed on both Steps 1 and 2 to determine the life expectancy on the equipment and catalysts used in the process.

3. A preliminary design for the integration of Steps 1 and 2 of the process should be developed, based on the larger-scale, longer-duration studies.

III. TECHNICAL DISCUSSION

Research on the first increment of Contract NAS 7-225 was initiated 22 April 1963 and completed 15 November 1963 (Reference 1). Research on the second increment of the contract was initiated on 16 November 1963 and completed on 15 July 1964. The latter program was divided into three tasks: Task 1, Design, Fabrication, and Testing; Task 2, Reduction of Igneous Rock; and Task 3, Reports.

A. BACKGROUND

Research conducted under the initial portion of Contract NAS 7-225 was devoted to a study of Step 2 of the Aerojet carbothermal process for the manufacture of oxygen from lunar minerals (see Equations 1 to 3). Research under Extension 1 of this contract was devoted to a study of Step 1 of the process (i.e., the reduction of silicate with methane and other reducing agents).

$$MgSiO_3 + 2\ CH_4 \longrightarrow 2\ CO + 4\ H_2 + Si + MgO \quad (1)$$

$$2\ CO + 6\ H_2 \longrightarrow 2\ CH_4 + 2\ H_2O \quad (2)$$

$$2\ H_2O \longrightarrow 2\ H_2 + O_2 \quad (3)$$

B. TASK 1, DESIGN, FABRICATION, AND TESTING

1. General Design

The equipment for the study of the reaction of methane with molten silicates was designed within the following guide lines: (a) induction heating, 450 kc, 10-kw maximum loading; (b) minimum reaction chamber volume to obtain best possible material balances using 0.25 lb of rock melt; (c) standard off-the-shelf items - particularly ceramic crucibles and tubes; (d) minimum use of glass for safety of operation; (e) minimum use of metal within the induction current field, except for the susceptor; and (f) adequate heat control around the reactor to permit the use of thermally sensitive bell jar seals.

Figure 1 is a schematic flow diagram of the silicate rock reduction furnace used on this program. The type of methane inlet used in the initial experiments is shown in this figure. Figure 2 is a simplified flow diagram showing the type of inlet tube and inlet tube cooling which were developed during the program.

Heat was supplied by means of induced electrical current, 450-kc frequency (2).* A radiometer head (3) controlled the temperature of the tungsten susceptor (4), which contained the rock melt in a ceramic crucible.

*Numbers refer to coding system used in Figure 1 to label reactor components.

Facilities were provided for evacuating the reactor and purging with argon before introducing the methane/hydrogen gases under positive reactor pressure (2 to 6 in. of water). Argon purge preceded shutdown. The vacuum pump (5) was usually idle, with the valve (6) closed to the system.

Fine regulation of gas flow was provided by manometers, pressure gages (PI-1, PI-2), and orifice plates (FI-1, FI-2) for methane (7) and hydrogen (8). A wet test meter (9) provided product gas measurement. The methane and hydrogen orifices were also calibrated for argon flow.

A gas chromatograph (10) monitored the carbon monoxide, hydrogen, and methane (if any) content of the exit gas. Water was extracted ahead of the chromatograph by drying tubes (11) - one tube for the water evolved during heatup, and one for water obtained during reduction. As carbon dioxide can be present only during heatup, a single Ascarite tube (12) is provided for its quantitative collection. A sample station (13) provided for mass spectrographic analysis, or a similar determination, to be performed at a different location.

The rock reactor was provided with temperature control, TC-1, by a radiometer (3) normally sighted at the mid-point of the susceptor wall. An optical pyrometer (14) provided temperature indication of the susceptor.

The rock reductions were initially studied by containing the melts in alumina (99.5% Al_2O_3, working temperature 1950°C) or zirconia crucibles. These crucibles were positioned inside the tungsten susceptor. Zirconia (16) insulation was provided between the tungsten susceptor and insulation retainer cylinder (17). The rock reactor was enclosed by a Pyrex bell jar (15). Cooling was provided by air streams through a base cooling ring (20) and a neck cooling ring (21), plus the water-cooled induction coil (19). A water condenser (22) cooled the product gases. The reactor was surrounded by shields during operation. Additional safety was provided by using metallic piping and valves (minimum use of glass), a pressure-relief device (23), and a flame arrester (24) on the outlet gas line.

The product gas temperature was measured at the reactor outlet (TI-2) and downstream of the cooler (TI-3). A mercury manometer (PI-3) measured reactor pressure. A second mercury manometer (not shown) was used to measure the pressure in the methane inlet tube.

Figures 3, 4, and 5 are photographs of the apparatus. Figure 3 shows the components of the reactor which have required the most critical design and fabrication. The assembled unit, in operating position in front of the Toccotron induction heater, is shown in Figure 4. The induction loading cell surrounds the quartz cylinder which, in turn, surrounds the tungsten susceptor. In the back of the shield is the radiometer head positioned to detect the tungsten susceptor wall temperature. There is a lower port so that the radiometer can be lowered to pick up the temperature of the mid-section of the susceptor. Ports in equivalent locations are provided for use of an optical pyrometer to sight the tungsten wall. Ports in the crucible cover, and in the heat shields shown in Figures 3 and 4, allow for views of the melt and susceptor.

Figure 5 is a photograph of the complete unit. On the left is the panel-board with manometers, orifices, and pressure gages for methane, hydrogen, and argon flow-control and measurement. In the center background is the Toccotron induction unit. The reactor and wet test meter are in the center foreground on the table. The product gas piping, the Drierite and Ascarite absorption tubes, the gas chromatograph, and the product gas sampling stations are located underneath the table. To the right are the recorders for the reactor temperature-controller and the gas chromatograph.

2. Fabrication of Rock Reduction Reactor

The reactor susceptor was fabricated from tungsten in the form of a 4-in.-high crucible with a 2-in. inside diameter and a 0.050-in. wall and bottom. These dimensions allow the use of standard-size ceramic crucibles, and provide annular space between the ceramic crucible and the susceptor (Figure 3).

The 4.5-in.-ID induction coil was fabricated from 0.75-in. by 0.25-in. rectangular copper tubing. Approximately 0.25-in. spacing was maintained between each of the five turns of the coil. The coil extended approximately 0.75-in. below, and 0.5 in. above, the susceptor (Figure 4).

A quartz cylinder was used to retain the refractory insulation in the annular space between the susceptor and the cylinder. A fire brick was shaped to provide separation of the quartz cylinder and the Pyrex base plate - the latter having holes for the induction furnace leads and outlet gas stream.

A standard 6-in. bell jar with a single 1-in. top inlet was used to enclose the reactor. A silicone grease seal was used on the ground surface of the Pyrex base. Bulkhead fittings with Teflon gaskets and Permatex gave vacuum-tight joints for passage of the induction coil leads and the outlet gas line.

3. Testing and Development

a. Equipment Operational Limits

Several tests were made to align the Toccotron with the rock reduction reactor to obtain the required furnace temperature with adequate control. The 450-kc induction heater brought the reactor to 1900°C within 90 min and maintained 1900°C ±10°C using the radiometer control. At this temperature, for as long as 4 hours, the insulating refractory retainer cylinder remained white and the bell jar surrounding the reactor remained below 70°C. During later runs the heating rate was kept to a maximum of 400°C per hour so that the crucible would not crack. This heating rate allows safe operation with a silicone grease seal to the Pyrex base and with a rubber top inlet seal for the feed gas lines. The Pyrex base was later replaced by one made from Transite after the Pyrex base broke repeatedly.

Ten runs were made. The runs in which major problems were encountered, and the modifications which were required, are presented in Table 1.

b. Toccotron

The 450-kc Toccotron was aligned with the susceptor load through the following steps (see Table 1, Runs 3, 4, 5, and 6): (1) removal of the Toccotron transformer to obtain 4000-v operation; (2) installation of new 3/8-in. copper induction coil leads having silver-soldered 2-in. by 1/16-in. flanges, separated by 1/16-in. Teflon strip; (3) elevation of the grid coil from within the tank coil to provide balanced loading; and (4) removal of some structural steel which picked up power from the induction field.

c. Reactor Heat Control

The reactor temperature was brought under control by the following steps: (1) replacement of quartz insulating refractory retaining cylinder with Fiberfrax cylinder (Table 1, Run 7); (2) use of a combination of fine and

coarse zirconia (Table 1, Run 8, same for Run 9 and 10); and (3) use of a ceramic cast susceptor heat shield plus secondary and tertiary Fiberfrax heat shields and Fiberfrax packing (Table 1, Runs 3, 4, and 7).

d. Temperature Control

The temperature control point was determined to be at the mid-point of the susceptor, or thereabouts, because the induction field within the bell jar prevented fogging at this point (Table 1, Run 9).

e. Susceptor

Early runs were made with a tungsten crucible as the susceptor. Later runs were made with a tungsten cylinder of equivalent wall dimensions with equally satisfactory performance, allowing reduced susceptor cost (Table 1, Run 9).

f. Auxiliary Apparatus

The gas metering equipment operated satisfactorily. The hydrogen and methane orifices were calibrated. In addition, these orifices were calibrated for argon flow. Argon was used as a supplementary gas for bringing the unit on stream, testing, and purging.

The gas chromatograph was calibrated for hydrogen, carbon monoxide, carbon dioxide, methane, argon, oxygen, and nitrogen. The wet test meter calibration was checked. The reactor operation pressure was about 0.2 in. of mercury.

g. Furnace Checkout with Basalt and Argon

A test was made to determine the operating characteristics of the furnace with basalt in the crucible. The reactor was charged with 53 g of basalt (4 to 30 mesh). The crucible temperature was slowly raised to 1167°C, while argon was metered into the reactor via the delivery tube with the alumina nozzle below the surface of the melt (0.25 in. above the bottom of the crucible).

h. Furnace Checkout with Quartz and Methane

Two runs were made with quartz melts. The first run (Table 1, Run 8) was made using an alumina crucible. Unfortunately, the sight ports became

fogged and it was feared that the reactor temperature had gone too high for the alumina crucible. No methane was admitted to the reactor. The second run was made using a zirconia crucible (Table 1, Run 9). This proved to be a very successful run insofar as the induction unit performance and its control are concerned. Near-perfect heat control of the reactor was achieved.

i. Inlet Tube Design

Throughout the 32 experiments, the inlet tube design evolved from (1) a single tube immersed into the melt to (2) a double tube with hydrogen cooling between the tubes to (3) a triple-walled tube with hydrogen flowing in the inner annulus and out the outer annulus to (4) a triple-walled tube terminating in a bell. The latter design is shown in Figure 6. The bell was a large (1-in. OD) tube with its open end extending to the bottom of the melt. The lower end of the bell was serrated to allow the carbon and hydrogen to bubble into the melt. The upper end was cemented with Zircoa Cast onto the triple-walled inlet tube. Alumina tubes were found to be entirely satisfactory for the triple-walled inlet (which was held up out of the melt and out of the hottest part of the furnace). Zirconia was the only material found which resisted attack by the melt; alumina and thoria were found to dissolve in the melt, and tungsten was found to be dissolved in the metal produced by the reduction of the silicate. The zirconia was found to react with carbon above about 1750°C.

j. Gas Flows

Argon gas was used in the bell jar to purge the product gas. The rate used was sufficient to purge the bell jar at least once every 15 min. An argon line was installed to purge gas around the crucible to keep the product gas (high in hydrogen) out of the hot zone. Cooling with hydrogen removed too much heat from the bell jar when the gas was discharged into the crucible or was allowed to flow through the hot zone. Keeping the hydrogen out of the bell jar by means of the triple-walled inlet tube solved this heat loss problem while providing inlet tube cooling.

C. TASK 2, REDUCTION OF IGNEOUS ROCK

1. Basis for Choice of Silicate Material

In the development of a chemical process for utilizing lunar raw materials, it is necessary to choose a material representative of the lunar surface

material. The mineral composition of an extremely small fraction of the lunar surface will be known after its examination by various probes and by the men of the Apollo missions; however, the detailed report of composition will have to await the establishment of lunar bases and geological exploration.

Insofar as the processing of lunar rock for the production of water and oxygen is concerned, it is useful to consider the bulk composition of lunar material. The gross composition of the Moon can probably be best represented by a mixture of metallic silicates with the iron in an unknown oxidation state (Reference 2). This conclusion is drawn from observational data and is consistent with cosmogenic theories of the solar system. Earth, Moon, and meteoritic matter may be thought to have the same general composition, except for iron content (Reference 2). The Moon does not appear to have an iron core; from its mean density of 3.34 g/cu cm, it may be concluded that only small percentages of free iron may be found. If the iron were removed from the Earth or from meteorites, the composition of these bodies would then more closely resemble that of igneous rock. In a recent paper, Kopal suggests that the Moon is of approximately the same composition as the terrestrial mantle (Reference 3). The terrestrial mantle has a much higher density (approximately 4.5 g/cu cm) than that of lunar rock; this may be accounted for by pressure-compression of presumed mantle rocks. Samples of mantle rock, however, are not available.

In choosing a terrestrial sample representative of lunar rock, crustal or igneous rock is a logical choice for two major reasons: (a) the composition resembles that of meteoritic material, and also the bulk of the Earth with iron removed; and (b) the density of igneous rock (3.3 g/cu cm) is close to the mean density of the Moon.

Representative lunar material may also be found among materials having extraterrestrial origin. A recent paper by Chapman and Larson (Reference 4) presents a basis for the belief that tektites have a lunar origin. If this is true, then either the lunar composition is high in silica, or material has been differentiated in the lunar crust with lighter, silica-rich, tektite material concentrated on the surface.

The naturally occurring materials used in the experiments described below were samples of granite and basalt, as representative of igneous rock, and Indochinites, as representative of tektites. Chemical analyses of the granite are given in Table 2. This granite was obtained in the Cactus Flats region of the San Bernardino mountains. Analyses of the basalt are given in Table 3A. This sample was obtained from the Pisgah Crater in San Bernardino County.

The Indochinites are from Thailand, and were purchased from the Wards Natural Science Establishment. The approximate range of composition of Indochinite tektites is shown in Table 3B (Reference 5).

2. Reporting Sequence for the Runs

A total of 32 runs was made during this contract period. The first ten runs were devoted to testing and adjusting the apparatus. Quantitative data were not obtained from all of the succeeding runs because of equipment failure. Runs which yielded useful quantitative data can be placed into three groups: (a) runs with carbon or silicon carbide, (b) runs with carbon and methane, and (c) runs with methane only. Improvement in equipment design occurred gradually throughout these runs, culminating in a run lasting over 7 hours with nearly quantitative carbon recovery.

Table 4 lists reaction conditions and results for Runs 11 through 32. No quantitative data were obtained from Runs 11, 12, and 13. In the first two runs, the Pyrex base failed and in Run 13 the methane inlet tube cracked when the methane flow was initiated. The need for the substitution of the Transite base plate and for the modification of the inlet tube design became readily apparent after these runs.

3. Runs Using Carbon or Silicon Carbide

a. Run 16

(1) Conditions

Run 16 was designed to react basalt with enough carbon to reduce all of the iron oxides and some of the silica to determine the temperatures and rates of the reduction reaction. A zirconia crucible was charged with basalt (50.1 g) and carbon (5.0 g) and placed in the reactor. An argon purge

(0.96 scfh) was used to sweep the product gases from the reactor. A record of the variables observed during the reaction is given in Table 5. The carbon monoxide content of the product gas gradually increased over a time period of 3.67 hours. At 1660°C, the reaction became very vigorous and a large amount of sublimate began to deposit on the induction coils; at this time the run was terminated.

(2) Results

The production of carbon monoxide occurring at the low temperatures was due to the reaction with iron oxides. This reaction became very vigorous at higher temperatures and caused a large amount of sublimate to be carried out of the melt. The amount of carbon recovered as carbon monoxide and carbon dioxide corresponded to 66% of the carbon in the charge (see Table 6). Examination of the reactor indicated that considerable frothing of the melt had occurred.

(3) Conclusions

The rate of temperature rise was too rapid to keep the reaction under control and obtain good carbon balances. Runs 17 through 32 were made using slower heating rates.

b. Run 17

(1) Conditions

A mixture of basalt (50.12 g) and carbon black (5.02 g) was placed in the silicate reactor, using a zirconia crucible. Tungsten wire (2.75 g) was placed in the crucible to test compatibility with the materials of the reaction. The argon purge was set at 0.96 scfh through the reactor, and the temperature was slowly raised. The variables during the run are given in Table 7.

The variation in temperature of the reactor, and in the carbon monoxide content of the product gas, is shown in Figure 7 where comparison with other runs can be made. The temperature was slowly raised to 1400°C over a period of 3.5 hours. During this time, carbon monoxide was evolved, giving a peak value of 3.33 mole % in the outlet gas before dropping to 1.60%. This temperature and rate of carbon monoxide evolution were maintained for 1.5 hours;

the temperature was then raised to 1520°C where the carbon monoxide evolution increased to 6.29%. After 1.5 hours, the carbon monoxide evolution again fell until the temperature was increased to 1670°C (at which temperature the carbon monoxide content rose to 17.1%). The temperature was raised to 1730°C, but the carbon monoxide content rapidly fell to less than 2% before the run was terminated.

(2) Results and Conclusions

The initial peaks of carbon monoxide evolution (Figure 7) represent reduction of iron oxide. The basalt sample contained 11.86 wt% of iron oxide (as Fe_2O_3) and would require 1.34 g of carbon if present as Fe_2O_3. The carbon monoxide evolved for the first 2.5 hours represents about 1.0 g of carbon. Other reducible materials in the basalt were titanium oxide (2.47% as TiO_2) and sodium oxide (3.73% as Na_2O). These oxides would consume 0.43 g of carbon. Consequently, only 35% of the carbon could have been oxidized by materials other than silica. The fact that 89.1% of the carbon was recovered as carbon monoxide indicates that a considerable portion of the silica of the sample was reduced at temperatures as low as 1550°C. Only a small amount of material (14.1 g) remained in the crucible; part of this was metal (7.38 g). Metal beads were also found on the inner side of the tungsten susceptor.

Analyses of the slag, metal, and sublimate are listed in Table 8. The slag contained 83% aluminum; the metal contained 66% iron, 13% tungsten, and 10% silicon. The sublimate contained 61% of the highly volatile sodium. The fact that the tungsten was dissolved in the metal indicates that tungsten crucibles cannot be used in this reaction.

c. Run 18

(1) Conditions

A mixture of granite (50.0 g) and carbon (5.0 g) contained in a zirconia crucible was placed in the reactor. With the argon purge set at 0.96 scfh, the temperature was slowly raised over a period of 8 hours. The variables during this time are given in Table 9. Figure 7 shows the variation of temperature and carbon monoxide percentage in the product gas. The experiment was continued until the carbon monoxide percentage dropped to nearly zero.

(2) Results and Conclusions

Much less carbon monoxide was produced at low temperatures (about 1000°C) than was obtained in Run 17. This is due to the lower percentages of reducible oxides in the granite which contained only small amounts of iron oxide (2.05% as Fe_2O_3), sodium oxide (3.10%), and potassium oxide (4.90%). These oxides would require 0.85 g of carbon for complete reduction (i.e., 17% of the initial carbon charge). Carbon oxides recovered account for 73% of the carbon; the silica reduction therefore accounts for most of the carbon monoxide evolved at 1550°C or higher. The slag material had non-magnetic pieces of metal dispersed throughout. The lack of a carbon balance could be due to reaction of silicon with carbon to form silicon carbide. An analysis of the slag showed that it contained 2.3% carbon (or nearly 20% of the carbon charge). Seven per cent of the carbon is unaccounted for. The results of this run show that reduction of silica in granite can occur at useful rates of temperatures as low as 1550°C.

d. Run 19

Run 19 was a repeat of Run 17, except that an alumina crucible was used. Data obtained during the run are given in Table 10. The rate of temperature rise is close to that for Run 17 but the final temperature reached was only 1612°C, compared to 1650°C in Run 17. The amount of carbon recovered (78%) is also less than that recovered in Run 17. Inspection of the alumina crucible after the run showed that it has sagged and broken. Due to the low final temperature and the broken crucible, only 78% of the carbon was recovered as carbon oxides.

e. Runs 20 and 21

Run 20 was a repeat of Run 18, except that a coarse-grained zirconia crucible and smaller quantities of reactants were used (42.0 g of granite and 4.2 g of carbon). The data obtained during the run are given in Table 11. A slightly higher temperature was reached at the end of Run 20 than was reached at the end of Run 18. The amount of carbon recovered as carbon monoxide is 67.6% compared to 65.3% in Run 18. The crucible and melt were recovered intact from this run. In Run 21 this crucible and its contents were reheated to 1770°C to recover more of the carbon as carbon monoxide. The data are given in Table 12. Additional carbon (0.49 g) was recovered as the oxide. The total amount of carbon

recovered from both runs is 88% of the charge of Run 20 (Table 6). The analysis of the metal produced in the reaction gave 64.9% iron and 19.5% silicon.

f. Run 22

(1) Conditions

A run was designed to determine the temperature and rate of the reaction between silicon carbide and granite. Because silicon carbide can easily form from silicon and carbon present in the reaction mixtures of the above runs, carbon losses would be realized if silicon carbide remained unreacted in the mixture. Granite (37.5 g) was mixed with finely divided silicon carbide (12.5 g) in a zirconia crucible and gradually heated in the reactor to 1738°C. The data obtained during this run are given in Table 13. Figure 7 shows the time rise in temperature and carbon monoxide content of the exit gas. Almost no reaction took place below 1100°C; about 7% of the reaction took place between 1100 and 1500°C. As the temperature was slowly increased from 1500 to 1738°C, the reaction rate gradually increased and then rapidly decreased as most of the carbon was consumed.

(2) Results and Conclusions

About 83% of the carbon in the silicon carbide was recovered as carbon monoxide and carbon dioxide (Table 6). Only a small amount of a dark-colored metallic-looking slag was left in the crucible. Although exact quantitative data could not be obtained, it was estimated that not more than 9% of the carbon was left in the slag. The rest was probably trapped in the crucible walls and in the crucible cover, both of which were noticeably blackened. The crucible was intact although it had been appreciably penetrated by carbon and other components of the melt. The analysis of the metal recovered from the melt gave 58.6% iron and 28.0% silicon as major constituents. Minor amounts of the following metals were found: Al, 4.5; Zr, 3.4; Ti, 2.0; Mg, 1.0; V, 1.0; Ni, 0.6; and Cu, 0.2. These results indicate that if silicon carbide is formed from a reaction between granite and carbon, an excess of granite will react with the carbide to produce silicon and carbon monoxide. The rate of this reaction is comparable to the production of carbon monoxide from granite and carbon.

4. Runs Using Carbon and Methane

a. Run 15

(1) Conditions

The failures of Runs 11, 12, and 13 pointed out the problem of maintaining a high reactor temperature when large quantities of hydrogen were passed into the reactor. In Run 15 the methane inlet tube was provided with an outer jacket to conduct cooling hydrogen around the inlet tube and out away from the heating zone. The inlet was fabricated from zirconia and alumina tubes. A zirconia crucible was charged with 99.1 g of basalt and 3.0 g of carbon. The reactor was heated with the following flows: argon purge (0.48 scfh), cooling H_2 (0.33 scfh), and inlet H_2 (0.205 scfh). At 1100°C, carbon monoxide evolution was first noted as the temperature was gradually raised; the evolution of carbon monoxide continued for 2.5 hours and then rapidly decreased. The maximum temperature which could be reached was 1590°C. A methane-hydrogen (95/5%) mixture was initiated at 0.1 scfh and maintained for 43 min. During this time the carbon monoxide content of the exit gas increased. The experiment was terminated when carbon began to accumulate on the induction coils. The conditions existing throughout the entire run are given in Table 14.

(2) Results

Inspection of the reactor showed that the inlet tube assembly had melted about 1/2-in. from the end but remained open to deliver methane above the melt surface. Magnetic metal (5.98 g) was recovered. The total carbon monoxide recovered corresponded to 3.17 g of carbon (i.e., 90% of the amount of carbon added to the charge).

(3) Conclusions

Hydrogen cooling of the inlet tube aided in keeping the inlet tube open. The fact that the reaction continued even after the inlet tube had melted at the tip, and had injected methane above the surface of the melt, suggested a new design for inlet tubes (Figure 6). This design was discussed above under Section III,A.

b. Run 23

(1) Conditions

This was the first run carried out with a triple-walled inlet tube terminating in a "bell." The outlet end of the inlet tube terminated 2.75-in. above the bottom of the crucible and about 2.25-in. above the surface of the molten rock. The charge was 70.95 g of basalt and 2.13 g of carbon. The reaction of the carbon and basalt was essentially complete (as indicated by a drop in CO content of product gas) by the time the crucible temperature reached 1550°C. The methane stream (94.4% CH_4 + 5.2% H_2) was then turned on at a rate of 0.032 scfh. The temperature was held constant at about 1550°C for 1.5 hours, at which time the methane line clogged. The conditions existing throughout the run are given in Table 15.

(2) Results and Conclusions

Post-run observations showed that the inlet tube cracked sometime during or prior to the run, allowing some of the methane to bypass the bell and the molten rock. A carbon balance showed that nearly all of the carbon mixed with the rock reacted to produce CO, but that a maximum of only 44% of the methane-carbon was converted to CO. It was indicated that the inlet tube must be raised further from the hot zone and/or the cooling hydrogen increased to prevent clogging of the inlet tube with hard carbon.

c. Run 24

(1) Conditions

The inlet tube was similar to the one for Run 23 except that the outlet end of the inlet tube was raised to 4.25-in. above the crucible bottom. The charge was 65.81 g of basalt plus 2.04 g of carbon contained in a 1.5 by 3-in. fine-grain zirconia crucible. The crucible was heated slowly to 1540°C (7.33 hours), allowing the carbon to react completely with the basalt. The methane (95.4% CH_4 + 5.2% H_2) was then turned on and maintained at 0.032 scfh for 3 hours, at which time the inlet tube clogged. A small amount of hydrogen (0.006 to 0.016 scfh) was mixed with the methane during this run. A cooling hydrogen flow of 0.38 to 0.52 scfh was passed through the inlet tube and then discharged into the bell

jar. This was the maximum hydrogen flow which could be used and still maintain the crucible temperature at 1540 to 1570°C. Data obtained for the run are given in Table 16.

(2) Results and Conclusions

Post-run inspection showed that the inlet tube and "bell" had functioned well; no methane had bypassed the molten rock. The carbon balance showed that although all of the carbon from the original charge had reacted to form CO, an average of only 50% of the methane-carbon had formed CO (a maximum of 80% of the methane-carbon formed CO for a 15-min period). Approximately one-half of the remaining carbon (0.32 g) was found deposited on the upper part of the inlet bell. It was concluded that either the inlet tube must be raised further up out of the hot zone, or the cooling hydrogen must be increased to prevent formation of hard carbon from clogging it. The slag was heated from Run 24 up to 1730°C with an argon purge to determine if there were carbon or carbides remaining in the slag which could be converted to CO at the higher temperatures. An insignificant amount of CO was formed.

d. Run 28

(1) Conditions

This run was carried out with a short "bell" (1-in. long, 1-in. OD) and a triple-walled inlet tube. The tip of the "bell" was not allowed to extend into the melt. The run lasted only 47 min after the methane flow was turned on. The charge was 70 g of basalt and 1.46 g of carbon.

(2) Results and Conclusions

A short "bell" lowers the inlet tube into the hot zone, thereby making it impossible to cool the methane sufficiently to prevent clogging of the inlet tube with carbon. No advantage was gained by delivering the methane above the melt surface. A carbon balance showed that almost all of the carbon mixed in the charge was converted to CO.

e. Run 29

(1) Description

A charge of 69.93 g basalt and 1.43 g carbon was used in a 1.5- by 3-in. impervious ZrO_2 crucible. A triple-walled inlet tube with a

1-in.-OD by 5-in.-long ZrO_2 "bell" was used. The charge was heated to 1565°C in 5 hours with 3.4 scfh cooling hydrogen discharging outside of the bell jar, and 0.9 scfh argon purge inside the bell jar. A methane flow of 0.031 scfh was maintained for 4.5 hours before the inlet tube clogged. Data obtained during the run are given in Table 17.

(2) Results and Conclusions

Increasing the length of the inlet "bell" from 4.5 to 5 in., and increasing the hydrogen cooling, greatly increased the length of the run before the inlet tube clogged. The carbon material balance showed that the maximum conversion of methane-carbon to carbon monoxide was 70%, but the average for the run was only 51%. Increasing the temperature from 1680 to 1700°C near the end of the run increased the conversion to CO from less than 50% to about 66%. Only about 25% of the missing carbon (the carbon not converted to oxides of carbon) was found as free carbon deposited in the inlet tube and bell. Some of the missing carbon had reacted with the top part of the inlet "bell" which had been completely penetrated with a dark gray-colored material.

5. Runs Using Methane Only

a. Run 14

(1) Conditions

A sample of granite (97.9 g) was placed in a zirconia crucible. A single 1/4-in. OD by 3/16-in. ID thoria inlet gas tube was used. A feed gas of 0.12 scfh of argon was maintained in the crucible throughout the run. In addition to the argon feed, an argon purge of 1.5 scfh was maintained in the bell jar throughout the run. The purpose of the argon purge was to sweep the products of combustion rapidly from the bell jar and to minimize the cooling effects of hydrogen. When the crucible temperature reached 1350°C, surging was noted in the manometer which measures the feed-gas-line pressure drop. This surging was believed to be caused by the argon gas bubbling through the molten granite. After about 5 min the bubbling stopped, indicating that the inlet tube had broken off above the liquid level. The Pyrex base cracked when the temperature reached 1690°C; because the crack was small, the run was continued. A flow of 0.06 scfh of 95%

CH_4 + 5% H_2 was fed through the inlet tube when the crucible reached 1800°C. After a few minutes a small amount of carbon was observed collecting on the bell jar. The methane rate was increased to 0.12 scfh after about 10 min. More carbon was noted on the bell jar, and after 15 min the inlet tube clogged. An analysis of the product gas showed that there was some carbon monoxide present, but quantitative data could not be obtained.

(2) Examination of the Reactor

The thoria inlet tube was found broken off 2-5/8-in. from the outlet end (i.e., about 1-1/2-in. above the liquid level). The broken piece of thoria could not be found in the melt. However, a thin white layer was noted on the bottom of the clear light-green glass. This white layer was believed to be the remains of the thoria tube which was dissolved by the glass. The crucible was intact but had leaked slightly through several small holes in its wall. The walls of the crucibles did not seem to have been bulged or thinned, so it is concluded that the leaks were caused by imperfection in the crucible. The portion of the inlet tube which remained in place was clogged with carbon formed by pyrolysis of the methane.

b. Run 26

(1) Conditions

The inlet tube for this run was fabricated from three concentric alumina tubes cemented to a 1/2-in. OD by 5-in.-long zirconia bell. The charge was 50.0 g of granite in a 1.5-in. by 3-in. fine-grain ZrO_2 crucible. The crucible was heated to 1550°C in about 7 hours with 0.016 scfh hydrogen flowing through the inlet tube. In addition, 1.4 scfh of hydrogen was flowing through the inlet tube cooling jacket, and 0.90 scfh purge argon was flowing into the bell jar. The methane stream was then maintained at 0.032 scfh while holding the temperature at 1550 to 1570°C. The other gas streams were maintained at constant rates: 1.4 scfh cooling hydrogen and 0.90 scfh argon flow. The methane flow was maintained for 3 hours until the CO concentration in the product gas had fallen so low that a very poor conversion of the methane was indicated. The inlet tube did not clog. Data obtained during the run are given in Table 18.

(2) Results and Conclusions

Post-run inspection showed that crucible, inlet tubes, and bell were intact. There was no sign of carbon in the melt or in the crucible. The inlet tube was free of carbon, but the inlet bell had carbon deposited on it - starting at the inlet end and diminishing in thickness until it disappeared an inch or so above the level of the melt inside of the bell. The carbon balances showed that a maximum of 64% of the methane-carbon was converted to CO some 45 min after the methane was turned on, but that the yield of CO dropped off to less than 40% within 1.5 hours after the run started. The small bell (1/2-in. instead of 1-in. OD) caused a more rapid buildup of carbon on the inside. Increasing the bell length from 4.5 to 5-in., increasing the hydrogen cooling, and mixing H_2 (1 part H_2 to 2 parts CH_4) with the methane prevented the deposition of carbon on the inlet tube.

c. Run 30

(1) Conditions

A charge of 70.0 g granite was used in a 1-1/2 by 3-in. impervious ZrO_2 crucible. A triple-walled inlet tube with a 1-in. by 5-in. ZrO_2 "bell" was used. The charge was heated to 1500°C in 4.5 hours with 4.5 scfh cooling hydrogen discharging outside the bell jar, and 0.93 scfh argon discharging inside the bell jar (the 0.93 scfh argon included 0.03 scfh argon flowing through the inlet tubes into the melt). A methane flow of 0.031 scfh was maintained for 3 hours before the inlet tube clogged. The crucible temperature was slowly raised during the methane injection from 1500 to 1700°C. Data obtained during the run are given in Table 19 and are shown graphically in Figure 8.

(2) Results and Conclusions

Seventeen-hundred degrees C seems to be the upper temperature limit with the 5-in.-long inlet bell, and the present hydrogen cooling of the methane line. Almost no carbon deposited in the crucible, in the bell jar, or in the drying tube. The top 1-in. of the zirconia inlet bell was darkened by carbon penetration. The inside of the bell contained hard carbon at the top (inlet end), which tapered off to a black shiny film at the level of the melt. Bell wall and crucible wall were both light gray at the melt level.

d. Run 31

(1) Conditions

A charge of 70.0 g of indochinite tektites was used in a 1.5-in. by 3-in. impervious ZrO_2 crucible. The inlet tube was identical in construction to the one used in Run 29. The charge was heated to 1555°C in 4 hours. A 3.4 scfh flow of cooling hydrogen was used during warmup, and a flow of 4.5 scfh to 6.0 scfh was used during methane reduction. An argon purge of 0.9 scfh was used throughout the run. The methane flow was initiated after the crucible temperature reached 1555°C at 0.030 scfh; after 1.0 hour, the rate was increased to 0.045 scfh; after 2 hours the rate was increased to 0.060 scfh. The methane flow was then terminated after 3 hours. The crucible temperature was slowly raised to 1610°C during the run. Data obtained during the run are given in Table 20 and shown in Figure 9.

(2) Results and Conclusions

Inspection showed that the inlet tube contained almost no carbon after 3 hours of operation at 1550 to 1610°C. The ceramic tubing seals on the inlet tube and bell were intact with no appreciable leakage. The bell and crucible were in excellent condition. The bell wall was only slightly darkened at the top. There was a rather thick layer of hard carbon inside the bell at the top, tapering off to a shiny black film 1.5-in. down. No carbon was in the crucible, and only a relatively small amount of carbon (36 mg) was in the bell jar and on the coil. Numerous small metal nodules were found in the slag (see Figure 10). Analysis of these nodules, which shows the presence of silicon, is given in Table 21. Extreme care was taken to obtain metal samples from the melt which were not contaminated with slag. The total carbon recovery was 99%. The results indicate that (a) the materials of construction were satisfactory for operation at 1600°C, and (b) while the inlet tube design could be used for many hours at 1600°C without clogging, its design would have to be modified to prevent deposition of hard carbon at the top of the bell.

e. Run 32

(1) Conditions

A charge of 70.0 g of basalt was used in a 1-1/2-in. by 3-in. impervious ZrO_2 crucible. The inlet tube was the same as for Runs 29, 30,

and 31, except that the bell was extended up 1.0 in. out of the hot zone (6-in. length). The charge was heated to 1555°C in 4.67 hours. A 4.0 scfh flow of cooling hydrogen was used during warmup, and a 6.0 scfh flow was used during methane reduction. An argon purge of 0.9 scfh was used throughout the run. The methane flow was maintained at 0.027 scfh for 2.0 hours, and was then increased to 0.043 scfh for the remainder of the run (5.67 hours). The run was terminated when carbon deposition on the bell jar allowed the current to arc from the heating coil to the glass bell jar. The crucible temperature was gradually raised during the run from 1555 to 1600°C. A very slow deposition of carbon on the bell jar and coil was noted shortly after the methane rate was increased. Data obtained during the run are given in Table 22 and are shown in Figure 11.

(2) Results and Conclusions

Inspection showed the inlet tube to be open, and only slightly black at outlet end. All ceramic seals were in excellent condition. The zirconia walls of the crucible and bell were light gray at the bottoms and dark gray at the top. The bell had a lyaer of hard carbon on the inside at the top, tapering down about 2 in. The slag was nearly white in color and contained metal nodules (see Figure 12). Once again, silicon was found on analysis of the metal (see Table 21); extreme care was taken to obtain slag-free metal samples.

The 6-in.-length bell, although permitting very long runs at 1600°C without clogging of the inlet tube, has not helped solve the problem of carbon deposition in the top part of the bell.

6. Material Balances

a. Carbon

The carbon balances obtained for the rock reactor are reported in two parts: (1) carbon balances for the carbon mixed with the rock charge, and (2) carbon recovered from the methane charged into the crucible.

Table 6 gives the carbon balances obtained for the carbon charged with the rock. The majority of this carbon was recovered as CO. In most cases, the amount of carbon recovered as CO_2 was 1% or less of that recovered as CO. The carbon recoveries were not as good as desired. In some runs, the crucibles

cracked or the molten rock bubbled over when the crucible temperature was raised too rapidly. Some of the carbon may have remained in the melt in some of the runs which were terminated at lower temperatures; at the higher temperatures, some of the carbon reacted with the crucible and "bell" materials. Several runs were made with basalt in which essentially 100% of the carbon was recovered (90 to 107%). Although the analytical and flow instruments were carefully calibrated many times, the experimental errors appear to be ±10%. This large error is due primarily to the very low flow rates of methane which had to be used. This flow was equivalent to about 1 inch of water using a fine orifice. This error was added to the inherent error of ±5% for gas chromatographic analysis. The recovery of carbon from the granite runs was somewhat lower (70 to 90%) as might be expected from the fact that this material has a higher melting point than basalt and was much more viscous when melted.

Table 23 gives carbon balances for three runs which were made with methane as the only source of carbon. In the last two runs, great care was taken to account for all of the carbon; the results account for 99% of the carbon. Although only 52 to 54% of the carbon was recovered as oxides of carbon, only a very small amount of carbon actually passed through the molten rock to the bell jar. This was very encouraging in view of the fact that the molten rock was only 0.25- to 1-in. deep.

b. Water Recovery

The data representing recovery of water from the exit gas stream are given for the most significant runs in Table 24. The data are quite scattered and there appears to be no correlation between the amount of water and the type or quantity of reactants used. Water may be produced in one of two ways: (1) by dehydration of the silicate, or (2) by the reduction of iron oxides with hydrogen (if both are present). In those runs using both carbon and methane, water could be produced in both ways. In either case, about 1 wt% of water (based on the amount of rock charged) is produced in this process.

The recovery of this quantity of water, although small, will enhance the attractiveness of the process because it represents a source of easily obtainable hydrogen.

IV. PERSONNEL

The senior staff assigned to this program was comprised of S. D. Rosenberg (Project Manager), G. A. Guter, F. E. Miller, and R. L. Beegle, Jr.

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TABLE 1

SUMMARY OF ROCK REDUCTION REACTOR IMPROVEMENTS

Run No.	Maximum Temperature Achieved °C	Major Problems Encountered	Modifications to Solve Problems
1	635	Induction unit tube burned out; it operated on half load.	Installed new tube.
2	974	Heat cracked bell jar neck.	Replaced bell jar and installed heat shield made of tungsten crucible cover, 1/8" alumina spacer, and 1/2" alumina dish filled with 14-28 mesh zirconia.
3	985	(a) Quartz cylinder, 1/8" extruded wall with drilled port holes for radiameter and optical pyrometer, broke. (b) Heat shield broke. (c) Induction heat was insufficient.	(a) Installed new cylinder of same material but without holes. (b-1) Cast 1-5/8" thick x 2-1/4" dia. zirconia heat shield with ports for top of tungsten crucible, Figure 5. (b-2) Cast secondary zirconia heat shield with ports and diameter of quartz cylinder, Figures 2 and 3. (c) Removed transformer.
4	1330	(a) Quartz cylinder broke. (b) Induction heat was insufficient. (c) Induction power heated (150°F) structural members near leads. (d) Bell jar was too hot considering susceptor temperature level.	(a) Installed new 1/4-in. wall cintered quartz cylinder, Figure 2. (b) Installed new induction furnace leads, 3/8" tubing, 2" x 1/16" silver soldered copper flanges with 1/16" Teflon separator. (c) No correction (d-1) Fiberfrax dome cover 1/2" thick, with quartz windows, fabricated (Figures 5 and 6). (d-2) In the 1-7/8" annular insulation space between susceptor and quartz cylinder, replaced outside 1/2-in. of 14-28 mesh zirconia with 325 mesh zirconia, GGC type.

TABLE 1 (Cont.)

Run No.	Maximum Temperature Achieved °C	Major Problems Encountered	Modifications to Solve Problems
5	1579	(a) Induction unit burned out bulbs, heat was insufficient. (b) Induction power heating steel adjacent to leads.	(a) Revised tank grid coil support so that it could be raised out of tank coil. (b) Installed copper shield.
6	1792	(a) Induction power continued to heat steel near leads. (b) Rear section of bell jar lower cooling-ring picked up induction field, causing heating and arcing. (c) Pyrex base plate broke due to Item b. (d) Too much heat came through quartz cylinder to bell jar wall.	(a) Removed steel and copper shield in areas of induction leads. (b) Replaced rear half of ring with plastic tubing drilled for air dispersion. (c) Installed new Pyrex base. (d) Installed molybdenum radiation shield midway within annular insulation space; alumina split-tube heat shield liner for quartz cylinder, Figure 2; 14-28 mesh zirconia refractory.
7	1502*	(a) Thermocouple at tip of gas injection nozzle picked up induction heat, Figures 2 and 3. (b) Quartz cylinder red, refractory passing too much heat. (c) Too much heat reaching upper portion of bell jar. (d) Basalt fogged quartz windows in heat shield.	(a) Removed thermocouple. (b) Replaced quartz cylinder with rigidized Fiberfrax shell, 1/16", and 4 layers of 1/16" Fiberfrax felt, Figures 5 and 8; replaced 14-28 mesh GGC zirconia with GGA, same mesh. (c) Used Fiberfrax blanket to pack between heat shields, Figure 6. (d) Windows cleaned.

* Maximum desired temperature for melt.

TABLE 1 (Cont.)

Run No.	Maximum Temperature Achieved °C	Major Problems Encountered	Modifications to Solve Problems
8	1822	(a) Milky deposit formed on shield windows; low temperature readings resulted.	(a) Windows easily cleaned for following run.
		(b) Alumina crucible melted, allowing molten quartz to run into tungsten crucible.	(b) Replaced tungsten crucible with new one; zirconia crucible used for next run.
		(c) Fiberfrax cylinder red; refractory passed more heat than desired for bell jar grease seal for long runs.	(c) Outside 1/2-in. of refractory space for length of susceptor filled with -48 + 150 mesh GGA zirconia, balance of area, next to susceptor -14 + 28 mesh.
9	1916**	(a) A major portion of the carbon released by bubbling methane into molten quartz passed out of the melt into bell jar, Figure 8.	
		(b) Carbon in upper portion of bell jar closed over windows for temperature reading, Figure 8.	(b) Moved optical pyrometer to lower port to sight on mid-section of susceptor.
		(c) Induction coil arced due to buildup of carbon on coil; Pyrex base cracked by arcing.	(c) Pyrex base replaced; inlet induction post insulated with Teflon.
		(d) Zirconia inlet jacket tube attacked by carbon, Figure 7.	(d) Use alumina tube for lower temperature melts, thoria 1900°C and above.
		(e) Zirconia crucible in one piece but fused to tungsten crucible.	(e) Position away from susceptor.
		(f) Tungsten crucible damaged in removal of zirconia discs for crucible support.	(f) Replaced with tungsten cylinder, same dimensions as tungsten crucible, and tungsten disc for bottom radiant heat shield.
10	1707*	Considerable carbon passed through melt into bell jar; inlet tubes plugged.	

** Fiberfrax cylinder shield remained white, showing excellent heat insulation. Bell jar top and bottom remained under 70°C. Induction furnace automatically controlled at ± 10°C; ample power reserve for higher future temperatures if desired.

* Maximum desired temperature for melt.

TABLE 2

ANALYSIS OF GRANITE

(Cactus Flats)

Chemical Analysis	
Constituent	Wt%
SiO_2	71.58
Al_2O_3	15.58
K_2O	4.90
Na_2O	3.10
Fe_2O_3	2.05
CaO	0.70
MgO	0.61
Loss at 105°C, H_2O	0.25
Loss between 105 and 600°C, H_2O	0.20

Spectrographic Analysis	
Constituent	Wt%
Si	28.
Al	11.
K	4.7
Na	2.1
Fe	1.4
Ca	0.42
Mg	0.23
Ti	0.14
Mn	0.043
Pb	0.022
Zr	0.021
Sr	0.011
V	0.0037
Ga	0.0036
Cu	0.0028
Cr	0.00075
Ba	trace
CO	trace
Ni	trace
Other elements	nil

TABLE 3A

ANALYSIS OF BASALT
(Pisgah Crater)

Chemical Analysis	
Constituent	Wt%
SiO_2	46.48
Al_2O_3	16.27
Fe_2O_3	11.86
CaO	9.05
MgO	8.50
Na_2O	3.73
TiO_2	2.47
SO_4	0.00
Loss at 105°C, H_2O	0.28
Loss between 105° and 550°C, H_2O	0.33

Spectrographic Analysis	
Constituent	Wt%
Si	27.
Al	11.
Fe	6.5
Ca	2.1
Mg	5.0
Na	1.2
Ti	1.3
Mn	0.13
Sr	0.042
Zr	0.022
V	0.018
Cr	0.016
Ni	0.010
Cu	0.0033
Ga	0.0034
CO	0.0031
Ba	trace
Other elements	nil

TABLE 3B

APPROXIMATE RANGE OF COMPOSITION OF INDOCHINITE TEKTITES

Constituent	Wt%
SiO_2	71.2 - 77.5
Al_2O_3	11.59 - 13.68
Fe_2O_3	0.37 - 0.82
FeO	3.25 - 4.88
MgO	1.62 - 2.96
CaO	1.57 - 2.08
Na_2O	1.06 - 1.58
K_2O	2.10 - 2.62
TiO_2	0.63 - 0.81
MnO	0.08 - 0.10

TABLE 4

CARBOTHERMAL REDUCTION OF NATURAL SILICATES
(Reaction Conditions and Results)

Run No.	Crucible Charge	Crucible	Inlet Tube	Inlet Gas	Maximum Temperature (°C)	Results
11	55.8 g granite	1-1/2" OD, 3-1/2" high, Al_2O_3	Single, 1/4" OD, Al_2O_3	argon; H_2	1800	Pyrex base cracked, crucible cracked, could not maintain 1800° with H_2 flow.
12	54.8 g granite	1-1/2" OD, 3" high ZrO_2	Double 1/4" ZrO_2, 1/8" Al_2O_3	H_2; argon	1800	Pyrex base cracked, crucible cracked, inlet tube broke.
13	72.9 g granite	1-1/2" OD, 3" high ZrO_2	Single 5/16" ZrO_2	argon; CH_4 + H_2 + A	1800	Inlet tube cracked off when CH_4 + H_2 was turned on, crucible OK, base OK.
14	97.9 g granite	1-1/2" OD, 3" high ZrO_2	Single 1/4" thoria	argon, CH_4 + H_2 + A	1800	Inlet tube broke off and dissolved in melt; inlet tube broke and plugged 5 min after CH_4 + H_2 turned on.
15	99.1 g basalt + 3.0 g carbon	1-1/2" OD, 3" high ZrO_2	Triple Al_2O_3 (H_2 cooled)	argon, H_2, CH_4 + H_2	1520	CO evolution started at 1100°C, CH_4 + H_2 flow for 43 min before inlet tube plugged. Iron was reduced in melt and some silica.
16	50.1 g basalt + 5.0 g carbon	1-1/2" OD, 3" high ZrO_2	None	argon purge	1670	Temperature increased too rapidly and frothing of melt occurred, white sublimate noted which deposited on bell jar; iron reduced;crucible intact; transite base OK.
17	50.1 g basalt + 5.0 g carbon	1-1/2" OD, 3" high ZrO_2	None	argon purge	1730	90% of carbon evolved as CO and CO_2. Considerable reaction at 1400-1500°C noted. Tungsten not compatible with melt; it dissolved in metal;most of material sublimed out of crucible.
18	50.0 g granite + 5.0 g carbon	1-1/2" OD, 3" high ZrO_2	None	argon purge	1646	73% C recovered as CO and CO_2, 20% C in melt, 7% C lost; crucible intact, but with grey discoloration. Slag contained droplets of metal. Appreciable quantities of white powder sublimed on bell jar.
19	50.0 g basalt + 5.0 g carbon	1-1/2" OD, 3-1/2" high Al_2O_3	None	argon purge	1645	Al_2O_3 crucible sagged and broke. Only 78% C recovered as CO and CO_2.
20	42.0 g granite + 4.2 g carbon	1-7/8" OD, 2" high ZrO_2	None	argon purge	1675	Used coarse grained ZrO_2 crucible with heavier wall; crucible in good shape. Only 72% C recovered as CO and CO_2.
21	Slag from Run 20	Same as Run 20	None	argon purge	1770	CO + CO_2 recovery increased to 88%; C in slag about 10%; crucible intact but badly corroded and attacked by melt.
22	37.5 g granite + 12.5 g SiC	1-7/8" OD, 2" high ZrO_2	None	argon purge	1755	83% of C recovered as CO + CO_2. Crucible intact but covered with flux. Most of melt had sublimed (only 12.4 g left). Slag contained most of missing C.

TABLE 4 (Cont.)

Run No.	Crucible Charge	Crucible	Inlet Tube	Inlet Gas	Maximum Temperature (°C)	Results
23	70.9 g basalt + 2.1 g carbon	1-1/2" OD, 3" high, Impervious ZrO_2	Triple walled with 1" x 4-1/2" ZrO_2 bell	94.4% CH_4 + 5.2% H_2	1550	Bell cracked and allowed CH_4 to escape reaction zone; 44% of CH_4 reacted to form CO
24	65.8 g basalt 2.0 g carbon	1-1/2" OD, 3" high, Fine grain ZrO_2	Triple walled with 1" x 4-1/2" ZrO_2 bell	94.4% CH_4 + 5.2% H_2	1540	Bell functioned well; 50% of methane reacted to form CO.
25	50.0 g granite	1-1/2" OD, 3" high, Fine grain ZrO_2	Triple walled with 1/2" x 5" ZrO_2 bell	94.4% CH_4 + 5.2% H_2	1520	CH_4 was inadvertently allowed to enter purge line and immediately cracked in bell jar; run aborted.
26	50.0 g granite	1-1/2" OD, 3" high, Fine grain ZrO_2	Triple walled with 1/2" x 5" ZrO_2 bell	94.4% CH_4 + 5.2% H_2	1570	CH_4 flow maintained for 3 hours; carbon deposited in inlet bell. Inlet tube did not clog.
27	Slag from Run 24	1-1/2" OD, 3" high, Fine grain ZrO_2	None	Argon purge	1730	An insignificant amount of CO was produced.
28	70 g basalt + 1.46 g carbon	1-1/2" OD, 3" high, Impervious ZrO_2	Triple walled with 1" x 7/8" impure ZrO_2 bell	94.4% CH_4 + 5.2% H_2	1565	Run lasted for 47 min before inlet tube clogged.
29	69.9 g basalt + 1.4 g carbon	1-1/2" OD, 3" high, Impervious ZrO_2	Triple walled with 1" x 5" bell	94.4% CH_4 + 5.2% H_2	1700	CH_4 flow maintained for 4.5 hours. Carbon appeared to react with inlet bell.
30	70.0 g granite	1-1/2" OD, 3" high, Impervious ZrO_2	Triple walled with 1" x 5" bell	94.4% CH_4 + 5.2% H_2	1700	CH_4 flow lasted for 3 hours before inlet clogged. Carbon deposited in inlet bell.
31	70.0 g tektite	1-1/2" OD, 3" high, Impervious ZrO_2	Triple walled with 1" x 5" bell	94.4% CH_4 + 5.2% H_2	1610	CH_4 flow maintained for 3 hours. Carbon deposited inside inlet bell. Inlet tube remained open.
32	70.0 g basalt	1-1/2" OD, 3" high, Impervious ZrO_2	Triple walled with 1" x 6" bell	94.4% CH_4 + 5.2% H_2	1600	CH_4 flow maintained for 7.67 hours. Carbon deposited in inlet bell. Inlet tube remained open.

TABLE 5

ROCK REACTOR DATA FOR RUN NO. 16*

Time	Crucible Temperature (°C)	Gas Flow Rates (scfh)			Carbon Monoxide Content in Product Gas (mole %)	Carbon Recovered as Carbon Monoxide (g C)
		Feed	Argon Purge	Product		
1:00	835	0	0.96	-	-	-
1:30	911	0	0.96	1.10	1.7	-
2:00	955	0	0.96	0.98	2.7	0.09
2:15	962	0	0.96	0.97	2.8	0.19
2:30	968	0	0.96	0.97	2.4	0.27
2:45	1055	0	0.96	1.03	3.3	0.39
3:00	1111	0	0.96	1.06	7.0	0.66
3:15	1165	0	0.96	1.04	7.0	0.92
3:30	1200	0	0.96	1.01	6.1	1.04
3:45	1272	0	0.96	1.05	5.7	1.25
4:00	1383	0	0.96	1.11	10.4	1.67
4:15	1463	0	0.96	1.08	10.7	2.05
4:30	1631	0	0.96	1.18	13.9	2.30
4:40	1660	0	0.96	1.27	21.9	2.96

* Inlet Tube - for argon purge 1/8" OD alumina
Crucible - 1-1/2" OD, 3" high impervious zirconia
Charge - 50.1 g basalt + 5.0 g carbon

TABLE 6

ROCK REACTOR CARBON BALANCES FOR REACTIONS IN WHICH CARBON WAS CHARGED WITH THE ROCK

Run No.	Rock Charged (g)	Maximum Temp (°C)	Carbon Charged (g)	Carbon Recovered			
				CO (g)	CO_2 (g)	Total (g)	%
15	Basalt (99.1)	1520	3.50	3.06	0.11	3.17	90
16	Basalt (50.1)	1670	5.00	2.96	0.35	3.31	66
17	Basalt (50.0)	1730	5.02	4.47	0.05	4.52	90
18	Granite (50.0)	1646	5.00	3.26	0.39	3.65	73
19	Basalt (50.0)	1645	5.00	3.19	0.69	3.88	78
21	Granite (42.0)	1770	4.20	3.34	0.36	3.70	88
22	Granite (37.5)	1755	3.75	2.99	0.10	3.09	83
23	Basalt (70.9)	1590	2.13	2.18	0.10	2.28	107
24	Basalt (65.8)	1570	2.04	2.00	0.10	2.10	103
29	Basalt (69.9)	1565	1.43	1.04	0.02	1.06	74

TABLE 7

ROCK REACTOR DATA FOR RUN NO. 17*

Time	Crucible Temperature (°C)	Gas Flow Rates (scfh) Feed	Argon Purge	Product	Carbon Monoxide Content in Product Gas (mole %)	Carbon Recovered as Carbon Monoxide (g C)
8:00	939	0	0.96	1.02	1.46	0.07
8:15	960	0	0.96	1.02	2.60	0.16
8:30	994	0	0.96	1.01	3.00	0.27
8:45	1020	0	0.96	1.01	2.96	0.38
9:00	1050	0	0.96	1.01	2.60	0.48
9:15	1112	0	0.96	1.02	2.80	0.57
9:30	1154	0	0.96	1.00	3.33	0.70
9:45	1185	0	0.96	0.99	2.40	0.79
10:00	1219	0	0.96	0.99	1.73	0.85
10:15	1300	0	0.96	1.00	1.94	0.92
10:30	1340	0	0.96	0.99	1.77	0.98
10:45	1361	0	0.96	1.00	1.77	1.04
11:00	1385	0	0.96	1.00	2.26	1.12
11:15	1400	0	0.96	1.00	2.13	1.20
11:30	1406	0	0.96	0.99	2.26	1.28
11:45	1410	0	0.96	1.00	1.60	1.34
12:00	1415	0	0.96	0.98	2.13	1.42
12:15	1420	0	0.96	0.99	1.86	1.48
12:30	1410	0	0.96	1.00	1.60	1.54
12:45	1488	0	0.96	1.00	2.00	1.61
1:00	1532	0	0.96	1.01	4.00	1.76
1:15	1515	0	0.96	1.01	4.15	1.91
1:30	1521	0	0.96	1.02	5.62	2.12
1:45	1528	0	0.96	1.05	4.40	2.28
2:00	1526	0	0.96	1.05	6.29	2.52
2:15	1526	0	0.96	1.03	5.13	2.71
2:30	1526	0	0.96	1.00	3.28	2.83
2:45	1523	0	0.96	0.99	2.46	2.91
3:00	1560	0	0.96	1.09	3.20	3.04
3:30	1640	0	0.96	0.91	11.10	3.98
4:00	1660	0	0.96	0.97	3.01	4.27
4:30	1650	0	0.96	1.00	1.86	4.47

* Inlet Tube - None
Crucible - 1-1/2" OD x 3" high impervious zirconia
Charge - 50.0 g basalt + 5.0 g carbon

TABLE 8

SPECTROGRAPHIC ANALYSES OF MATERIALS FROM ROCK REACTOR
(wt% ± 25%)

Run No.	Sample	Na	Si	Ga	Al	Zr	Fe	Mg	Cu	Ca	Ti	Ni	W	V
17	Sublimate	61.0	13.8	11.1	5.5	5.5	1.1	1.6	2.7	-	-	-	-	-
18	Sublimate	55.0	41.0	0.5	2.7	-	-	0.5	0.0	-	-	-	-	-
19	Sublimate	63.1	23.4	6.0	2.9	1.8	0.9	1.4	0.2	-	-	-	-	-
17	Slag	-	1.5	-	83.0	9.2	-	2.3	-	1.6	2.3	-	-	-
18	Slag	39.0	39.0	-	12.0	6.1	-	2.4	-	1.2	-	-	-	-
21	Slag	23.5	8.2	-	62.9	2.5	1.3	0.5		0.6	0.5	-	-	-
22	Slag	-	18.6	-	51.8	14.0	2.5	0.8	-	5.9	6.7	-	-	-
17	Metal	-	10.5	-	2.2	0.4	66.1	0.2	0.2	-	2.2	1.1	13.2	3.9
18	Metal	-	7.8	-	2.9	4.8	77.6	0.6	0.5	-	2.9	0.004	-	2.5
21	Metal	-	19.5	-	2.2	7.6	64.9	0.3	0.1	-	2.2	1.1	-	2.2
22	Metal	-	28.0	-	4.5	3.4	58.6	1.0	0.2	-	2.0	0.6	-	1.0

TABLE 9

ROCK REACTOR DATA FOR RUN NO. 18*

Time	Crucible Temperature (°C)	Gas Flow Rates (scfh)			Carbon Monoxide Content in Product Gas (mole %)	Carbon Recovered as Carbon Monoxide (g C)
		Feed	Argon Purge	Product		
8:15	852	0	0.96	0.97	0.0	0.00
8:30	934	0	0.96	0.97	0.2	0.01
8:45	977	0	0.96	0.97	0.2	0.02
9:00	1032	0	0.96	0.97	0.5	0.03
9:15	1050	0	0.96	0.97	0.6	0.05
9:30	1070	0	0.96	0.97	0.4	0.07
9:45	1105	0	0.96	0.97	0.3	0.02
10:00	1170	0	0.96	0.97	-	0.09
10:15	1205	0	0.96	0.98	1.2	0.11
10:30	1288	0	0.96	0.98	1.1	0.15
10:45	1300	0	0.96	0.97	0.9	0.18
11:00	1333	0	0.96	0.97	0.9	0.21
11:15	1350	0	0.96	0.97	0.9	0.24
11:30	1372	0	0.96	0.97	0.7	0.27
11:45	1400	0	0.96	0.97	0.9	0.30
12:00	1455	0	0.96	0.98	1.7	0.36
12:15	1444	0	0.96	0.98	1.9	0.43
12:30	1461	0	0.96	0.99	2.1	0.50
12:45	1488	0	0.96	0.99	2.6	0.60
1:00	1505	0	0.96	0.99	2.7	0.69
1:15	1516	0	0.96	1.00	3.4	0.81
1:30	1540	0	0.96	1.02	5.9	1.03
1:45	1540	0	0.96	1.04	7.2	1.30
2:00	1540	0	0.96	1.05	8.0	1.60
2:15	1560	0	0.96	1.06	9.1	1.94
2:30	1560	0	0.96	1.04	6.9	2.20
2:45	1560	0	0.96	1.03	5.8	2.41
3:00	1600	0	0.96	1.06	8.8	2.75
3:15	1620	0	0.96	1.06	9.4	3.10
3:30	1640	0	0.96	0.99	2.0	3.17
3:45	1646	0	0.96	0.98	1.7	3.23
4:00	1646	0	0.96	0.97	0.7	3.26

* Inlet Tube - None
Crucible - 1-1/2" OD x 3" high impervious zirconia
Charge - 50.0 g granite + 5.0 g carbon

TABLE 10

ROCK REACTOR DATA FOR RUN NO. 19*

Time	Crucible Temperature (°C)	Gas Flow Rates (scfh)			Carbon Monoxide Content in Product Gas (mole %)	Carbon Recovered as Carbon Monoxide (g C)
		Feed	Argon Purge	Product		
8:00	900	0	0.96	0.98	0.1	0.00
8:15	950	0	0.96	0.99	0.4	0.01
8:30	963	0	0.96	0.99	1.1	0.06
8:45	1015	0	0.96	0.99	1.7	0.12
9:00	1050	0	0.96	0.99	2.0	0.19
9:15	1067	0	0.96	0.99	1.8	0.25
9:30	1082	0	0.96	0.99	1.5	0.30
9:45	1088	0	0.96	0.99	1.2	0.35
10:00	1110	0	0.96	0.99	1.6	0.40
10:15	1132	0	0.96	0.98	2.0	0.47
10:30	1136	0	0.96	0.98	1.6	0.53
10:45	1218	0	0.96	0.98	1.4	0.57
11:00	1263	0	0.96	0.98	2.3	0.66
11:15	1296	0	0.96	0.98	2.0	0.73
11:30	1331	0	0.96	0.98	1.5	0.78
11:45	-	0	0.96	0.97	0.8	0.81
12:00	1260	0	0.96	0.97	0.1	0.81
12:15	1320	0	0.96	0.97	0.2	0.82
12:30	1348	0	0.96	0.97	0.3	0.83
12:45	1360	0	0.96	0.97	0.6	0.85
1:00	1428	0	0.96	0.98	1.0	0.89
1:15	1467	0	0.96	0.98	1.3	0.93
1:30	1477	0	0.96	0.98	2.0	1.00
1:45	1482	0	0.96	0.99	2.0	1.07
2:00	1526	0	0.96	1.00	1.8	1.14
2:15	1550	0	0.96	1.06	4.6	1.31
2:30	1560	0	0.96	1.07	7.1	1.59
2:45	1557	0	0.96	1.06	4.9	1.77
3:00	1552	0	0.96	1.01	5.7	1.98
3:15	1580	0	0.96	1.07	5.3	2.19
3:30	1610	0	0.96	1.08	8.7	2.53
3:45	1640	0	0.96	1.03	6.4	2.76
4:00	1642	0	0.96	1.00	5.2	2.95
4:15	1630	0	0.96	0.99	4.4	3.11
4:30	1612	0	0.96	0.99	2.4	3.19

* Inlet Tube - None
Crucible - 1-1/2" OD x 3-1/2" high alumina
Charge - 50.0 g basalt + 5.0 g carbon

TABLE 11

ROCK REACTOR DATA FOR RUN NO. 20*

Time	Crucible Temperature (°C)	Gas Flow Rates (scfh)			Carbon Monoxide Content in Product Gas (mole %)	Carbon Recovered as Carbon Monoxide (g C)
		Feed	Argon Purge	Product		
8:45	935	0	0.96	0.99	0.2	0.01
9:00	1000	0	0.96	0.99	0.6	0.03
9:15	1052	0	0.96	0.99	0.8	0.05
9:30	1055	0	0.96	0.99	0.7	0.08
9:45	1094	0	0.96	0.99	0.7	0.11
10:00	1116	0	0.96	0.98	0.6	0.13
10:15	1128	0	0.96	0.98	0.7	0.16
10:30	1162	0	0.96	0.97	0.7	0.18
10:45	1192	0	0.96	0.97	0.8	0.20
11:00	1250	0	0.96	0.97	0.8	0.23
11:15	1278	0	0.96	0.97	0.7	0.26
11:30	1290	0	0.96	0.97	0.7	0.28
11:45	1342	0	0.96	0.97	0.7	0.31
12:00	1362	0	0.96	0.97	1.1	0.35
12:15	1395	0	0.96	0.98	1.1	0.39
12:30	1410	0	0.96	0.99	1.2	0.43
12:45	1436	0	0.96	0.99	2.5	0.51
1:00	1476	0	0.96	1.03	2.7	0.61
1:15	1495	0	0.96	1.03	5.8	0.83
1:30	1505	0	0.96	1.03	5.8	1.04
1:45	1520	0	0.96	1.03	5.9	1.26
2:00	1532	0	0.96	1.02	5.8	1.47
2:15	1546	0	0.96	1.06	5.7	1.69
2:30	1570	0	0.96	1.04	8.5	2.01
2:45	1588	0	0.96	1.02	7.7	2.30
3:00	1583	0	0.96	1.01	4.8	2.47
3:15	1590	0	0.96	0.99	3.2	2.58
3:30	1615	0	0.96	0.99	2.1	2.66
3:45	1640	0	0.96	0.98	2.1	2.73
4:00	1640	0	0.96	0.97	1.2	2.78
4:15	1660	0	0.96	0.98	0.8	2.80
4:30	1670	0	0.96	0.97	1.0	2.84

* Inlet Tube - None
Crucible - 1-7/8" OD x 2" high zirconia
Charge - 42.0 g granite + 4.2 g carbon

TABLE 12

ROCK REACTOR DATA FOR RUN NO. 21*

Time	Crucible Temperature (°C)	Gas Flow Rates (scfh) Feed	Argon Purge	Product	Carbon Monoxide Content in Product Gas (mole %)	Carbon Recovered as Carbon Monoxide (g C) **
11:30	910	0	0.96	0.96	0.0	2.85
12:00	1115	0	0.96	0.96	0.0	2.85
12:30	1342	0	0.96	0.96	0.0	2.85
12:45	1405	0	0.96	0.96	0.1	2.85
1:00	1460	0	0.96	0.96	0.1	2.85
1:15	1505	0	0.96	0.97	0.1	2.85
1:30	1560	0	0.96	0.97	0.1	2.86
1:45	1572	0	0.96	0.97	0.2	2.86
2:00	1620	0	0.96	0.97	0.3	2.87
2:15	1650	0	0.96	0.97	0.4	2.89
2:30	1660	0	0.96	0.96	0.5	2.90
2:45	1700	0	0.96	0.96	1.0	2.94
3:00	1730	0	0.96	0.97	1.5	2.99
3:15	1735	0	0.96	0.97	1.6	3.05
3:30	1755	0	0.96	0.97	1.6	3.11
3:45	1765	0	0.96	0.97	1.8	3.17
4:00	1765	0	0.96	0.97	1.6	3.23
4:15	1765	0	0.96	0.97	1.6	3.21
4:30	1770	0	0.96	0.98	1.6	3.34

* Inlet Tube - None
Crucible - Same as Run No. 20
Charge - Slag from Run No. 20

** Cumulative including Run No. 20

TABLE 13

ROCK REACTOR DATA FOR RUN NO. 22*

Time	Crucible Temperature (°C)	Gas Flow Rates (scfh) Feed	Argon Purge	Product	Carbon Monoxide Content in Product Gas (mole %)	Carbon Recovered as Carbon Monoxide (g C)
7:15	1015	0	0.96	1.03	0.1	0.00
7:45	1280	0	0.96	1.00	0.9	0.08
8:15	1412	0	0.96	0.97	0.4	0.10
8:45	1490	0	0.96	0.97	1.2	0.18
9:00	1505	0	0.96	0.97	1.1	0.22
9:15	1515	0	0.96	0.97	1.1	0.26
9:30	1540	0	0.96	0.97	1.5	0.31
9:45	1546	0	0.96	0.97	1.3	0.36
10:00	1580	0	0.96	0.97	1.4	0.40
10:15	1592	0	0.96	0.97	1.6	0.46
10:30	1610	0	0.96	0.97	1.1	0.50
10:45	1622	0	0.96	0.97	1.1	0.53
11:00	1633	0	0.96	0.98	1.6	0.59
11:15	1643	0	0.96	0.98	2.4	0.67
11:30	1700	0	0.96	0.98	2.3	0.76
11:45	1633	0	0.96	0.98	2.1	0.83
12:00	1642	0	0.96	0.97	2.3	0.91
12:15	1676	0	0.96	0.98	3.2	1.03
12:30	1670	0	0.96	0.99	3.4	1.14
12:45	1670	0	0.96	0.99	3.3	1.26
1:00	1660	0	0.96	0.98	2.8	1.36
1:15	1650	0	0.96	0.99	2.3	1.45
1:30	1670	0	0.96	0.99	2.3	1.53
1:45	1690	0	0.96	0.99	3.4	1.65
2:00	1685	0	0.96	0.99	3.6	1.78
2:15	1695	0	0.96	0.97	3.7	1.91
2:30	1710	0	0.96	1.01	4.4	2.07
2:45	1715	0	0.96	1.00	5.2	2.26
3:00	1715	0	0.96	0.99	4.4	2.41
3:15	1715	0	0.96	0.99	3.8	2.55
3:30	1715	0	0.96	0.99	3.3	2.67
3:45	1720	0	0.96	0.99	3.0	2.77
4:00	1720	0	0.96	0.99	2.6	2.86
4:15	1720	0	0.96	0.98	2.2	2.94
4:30	1738	0	0.96	0.97	1.5	2.99

* Inlet Tube - None
Crucible - 1-7/8" OD x 2" high zirconia
Charge - 37.5 g granite + 12.5 g SiC

TABLE 14

ROCK REACTOR DATA FOR RUN NO. 15*

Time	Crucible Temperature (°C)	Gas Flow Rates (scfh) CH_4 Feed	H_2 Feed	H_2 Cooling	Argon Purge	Product	Carbon Monoxide Content in Product Gas (mole %)	Carbon Recovered as Carbon Monoxide (g C)
1:00	883	0	0.20	0.32	0.48	1.0	6.0	0.21
1:15	1098	0	0.20	0.32	0.48	1.07	16.0	0.82
1:30	1143	0	0.20	0.32	0.48	1.04	11.5	1.25
1:45	1141	0	0.20	0.32	0.48	1.02	8.4	1.75
2:00	1146	0	0.20	0.32	0.41	1.00	5.3	1.86
2:15	1192	0	0.20	0.32	0.48	0.96	3.2	2.07
2:30	1250	0	0.20	0.32	0.41	0.96	6.0	2.27
2:45	1250	0	0.20	0.32	0.48	0.98	5.7	2.41
3:00	1316	0	0.20	0.07	0.48	0.93	4.4	2.56
3:15	1480	0	0.21	0.07	0.48	0.75	5.5	2.67
3:30	1522	0	0.20	0.07	0.48	0.62	4.6	2.70
3:45	1570	0	0.20	0.07	0.48	0.61	1.2	2.71
4:00	1590	0.050	0.20	0.07	0.48	0.71	0.1	2.71
4:15	1570	0.050	0.20	0.07	0.48	0.75	4.3	0.07**
4:30	1550	0.050	0.20	0.07	0.48	0.75	5.1	0.18
4:45	1550	0.050	0.20	0.07	0.48	0.70	4.6	0.35

* Inlet Tube - triple wall Al_2O_3 tubes
Crucible - 1-1/2" OD, 3" high impervious zirconia
Charge - 99.1 g basalt + 3.0 g carbon

** carbon from methane

TABLE 15

ROCK REACTOR DATA FOR RUN 23*

Time	Crucible Temp (°C)	Gas Flow Rates (scfh)				Carbon Monoxide Content in Product Gas (mole %)	Carbon Recovered as Carbon Monoxide (g C)
		Methane Feed	Argon Purge	H_2 Cooling	Product Gas		
9:05	1030	0	0.90	0.18	1.15	9.4	0.39
9:15	1060	0	0.90	0.18	1.14	5.8	0.63
9:30	1070	0	0.90	0.18	1.13	3.7	0.78
9:45	1085	0	0.90	0.18	1.12	2.6	0.88
10:00	1140	0	0.90	0.18	1.12	2.8	1.00
10:15	1195	0	0.90	0.18	1.13	2.8	1.11
10:30	1305	0	0.90	0.18	1.12	2.7	1.21
10:45	1390	0	0.90	0.18	1.15	1.6	1.28
11:00	1440	0	0.90	0.18	1.12	2.0	1.37
11:15	1470	0	0.90	0.18	1.13	2.7	1.47
11:30	1510	0	0.90	0.18	1.13	2.9	1.58
11:45	1500	0	0.90	0.52	1.39	3.5	1.76
12:00	1470	0	0.90	0.52	1.41	2.6	1.90
12:15	1530	0	0.90	0.52	1.41	2.3	2.02
12:30	1570	0	0.90	0.52	1.39	1.1	2.07
12:45	1530	0	0.90	0.74	1.63	0.5	2.10
1:00	1520	0	0.90	0.74	1.64	0.2	2.11
1:15	1535	0	0.90	0.74	1.63	0.2	2.12
1:30	1540	0	0.90	0.74	1.63	0.1	2.13
2:00	1560	0	0.90	0.74	1.81	0.5	2.16
2:15	1545	0	0.90	0.74	1.75	0.2	2.17
2:30	1560	0.032	0.90	0.74	1.66	0.1	2.18
2:45	1570	0.032	0.90	0.74	1.66	0.7	0.04
3:00	1550	0.032	0.90	0.74	1.65	0.8	0.09
3:15	1550	0.032	0.90	0.74	1.66	0.7	0.13
3:30	1549	0.032	0.90	0.74	1.66	0.6	0.18
3:45	1547	0.032	0.90	0.74	1.66	0.2	0.19
4:00	1555	0.032	0.90	0.74	1.59	0.4	0.21

*Inlet Tube - triple-walled alumina tubes terminating in a 1-in.-OD by 4.5-in.-long ZrO_2 "bell." Outlet of alumina tube 2-3/4-in. above the bottom of the crucible. Crucible - 1-1/2-in. by 3-in. deep impervious ZrO_2. Charge - 7095 g of basalt mixed with 2.13 g of carbon. CO_2 recovered = 0.38 g.

TABLE 16

ROCK REACTOR DATA FOR RUN 24*

Time	Crucible Temp (°C)	Gas Flow Rates (scfh)				Carbon Monoxide Content in Product Gas (mole %)	Carbon Recovered as Carbon Monoxide (g C)
		Methane Feed	Argon Purge	H_2 Cooling	Product Gas		
12:15	760	0	0.33	1.40	1.67	-	-
12:30	800	0	0.33	1.40	1.67	0.1	0.01
1:00	950	0	0.33	1.40	1.72	1.8	0.13
1:30	1030	0	0.33	1.40	1.68	2.1	0.37
2:00	1100	0	0.33	1.40	1.76	1.9	0.59
2:30	1180	0	0.33	1.40	1.72	3.6	0.99
3:00	1250	0	0.33	0.75	1.08	2.3	1.23
3:30	1365	0	0.33	0.75	1.05	5.6	1.65
4:00	1430	0	0.33	0.75	1.01	2.1	1.85
4:30	1490	0	0.33	0.52	0.82	0.9	1.95
5:00	1550	0.032	0.33	0.52	0.78	0.8	2.00
5:15	1555	0.032	0.33	0.52	0.83	0.8	0.02
5:30	1540	0.032	0.33	0.52	0.87	2.8	0.11
5:45	1530	0.032	0.33	0.52	0.87	2.2	0.18
6:00	1515	0.032	0.33	0.52	0.86	1.8	0.24
6:15	1512	0.032	0.33	0.52	0.85	1.9	0.30
6:30	1520	0.032	0.33	0.52	0.85	1.4	0.34
6:45	1522	0.032	0.48	0.20	0.90	1.4	0.38
7:00	1530	0.032	0.48	0.20	0.92	2.1	0.45
7:15	1510	0.032	0.48	0.20	0.90	2.1	0.52
7:30	1520	0.032	0.48	0.20	0.91	1.4	0.57
7:45	1515	0.032	0.48	0.20	0.91	1.3	0.61
8:00	1515	0	0.48	0.20	0.84	1.2	0.65
8:15	-	0	0.48	0.20	-	1.2	0.68
8:30	1530	0	0.48	0.20	0.84	0.6	0.71
8:45	1650	0	0.48	0.20	0.90	0.6	0.73
9:00	1740	0	0.90	0.20	-	2.1	0.79

*Inlet Tube - Same as Run 23 except that the end of the triple-walled inlet tube was raised approximately 2 in. so that it was 4-1/4 in. above the bottom of the crucible and only 1/4-in. below the closed end of the "bell." Crucible - 1-1/2-in. x 3-in. deep regular fine-grain ZrO_2. Charge - 65.81 g basalt mixed with 2.04 g carbon. CO_2 recovered = 0.37 g.

TABLE 17

ROCK REACTOR DATA FOR RUN 29*

Time	Crucible Temp (°C)	Gas Flow Rates (scfh)				Carbon Monoxide Content in Product Gas (mole %)	Carbon Recovered as Carbon Monoxide (g C)
		Methane Feed	Argon Purge	H_2 Cooling	Product Gas		
9:15	935	0	0.90	3.4	1.00	0.2	0.01
9:30	1005	0	0.90	3.4	1.02	2.9	0.11
10:00	1110	0	0.90	3.4	0.99	4.3	0.44
10:30	1200	0	0.90	3.4	0.94	2.6	0.68
11:00	1310	0	0.90	3.4	0.94	1.2	0.77
11:30	1400	0	0.90	3.4	0.94	1.8	0.87
12:00	1450	0	0.90	3.4	0.94	0.9	0.95
12:30	1525	0	0.90	3.4	0.93	0.8	1.02
1:00	1565	0.028	0.90	3.4	0.93	0.2	1.04
1:15	1580	0.031	0.90	3.4	0.94	0.1	0.01
1:30	1575	0.031	0.90	3.4	0.97	1.5	0.06
1:45	1565	0.031	0.90	3.4	0.97	2.2	0.14
2:00	1565	0.031	0.90	3.4	0.97	2.0	0.21
2:15	1580	0.031	0.90	3.4	0.97	1.9	0.27
2:30	1600	0.031	0.90	3.4	0.97	1.7	0.33
2:45	1600	0.031	0.90	3.4	0.97	0.9	0.36
3:00	1610	0.031	0.90	3.4	0.97	1.7	0.42
3:15	1615	0.031	0.90	3.4	0.97	1.6	0.48
3:30	1610	0.031	0.90	3.4	0.97	1.6	0.54
3:45	1615	0.031	0.90	3.4	0.97	0.8	0.56
4:00	1615	0.031	0.90	3.4	0.97	1.5	0.62
4:15	1630	0.031	0.90	3.4	0 97	1.0	0.65
4:30	1635	0.031	0.90	3.4	0.97	1.6	0.71
4:45	1660	0.031	0.90	3.4	0.98	1.8	0.77
5:00	1685	0.031	0.90	3.4	0.98	2.0	0.84
5:15	1700	0.031	0.90	3.4	0.97	2.0	0.92
5:30	1695	0.031	0.90	3.4	1.01	0.9	0.95
5:45	1690	0	0.90	3.4	0.90	0.4	0.96

*Inlet Tube - triple-walled alumina tube terminating in a 1-in.-OD by 5-in.-long ZrO_2 "bell." Crucible - 1-1/2-in. by 3-in. deep impervious ZrO_2. Charge - 69.93 g of basalt mixed with 1.43 g carbon. CO_2 recovered = 0.06 g; H_2O recovered = 1.15 g.

TABLE 18

ROCK REACTOR DATA FOR RUN 26*

Time	Crucible Temp (°C)	Gas Flow Rates (scfh)				Carbon Monoxide Content in Product Gas (mole %)	Carbon Recovered as Carbon Monoxide (g C)
		Methane Feed	Argon Purge	H_2 Cooling	Product Gas		
8:00	800	0	0.9	0.75	-	-	-
8:30	970	0	0.9	0.75	-	-	-
9:00	1050	0	0.9	0.52	-	-	-
9:30	1150	0	0.9	0.52	-	-	-
10:00	1210	0	0.9	0.52	-	-	-
10:30	1330	0	0.9	0.52	-	-	-
11:00	1400	0	0.9	0.52	-	-	-
11:30	1450	0	0.9	0.52	-	-	-
12:00	1520	0	0.9	0.52	-	-	-
12:30	1540	0	0.9	1.40	-	-	-
1:00	1550	0	0.9	1.2	-	0	-
1:15	1540	0.032	0.9	1.20	1.90	0	0
1:30	1550	0.032	0.9	1.10	1.97	0.8	0.06
1:45	1548	0.032	0.9	1.40	2.08	0.9	0.13
2:00	1550	0.032	0.9	1.40	2.08	0.9	0.19
2:15	1540	0.032	0.9	1.40	2.08	0.4	0.22
2:30	1530	0.032	0 9	1.20	1.88	0.6	0.27
2:45	Explosion in nearby apparatus caused evacuation of laboratory. Run aborted.						

*Inlet Tube - triple-walled alumina tubes terminating in a 1/2-in.-OD by 5-in.-long ZrO_2 "bell." Crucible - 1-1/2-in. by 3-in.-deep standard fine-grain ZrO_2 crucible. Charge - 50.0 g of granite (no carbon).

TABLE 19

ROCK REACTOR DATA FOR RUN 30*

Time	Crucible Temp (°C)	Gas Flow Rates (scfh)				Carbon Monoxide Content in Product Gas (mole %)	Carbon Recovered as Carbon Monoxide (g C)	Methane Converted to Carbon Monoxide (mole %)
		Methane Feed	Argon Purge	H_2 Cooling	Product Gas			
9:15	915	0	0.90	4.5	0.57	0	0	-
9:30	985	0	0.90	4.5	-	0	0	-
10:00	1060	0	0.90	4.5	0.90	0	0	-
10:30	1178	0	0.90	4.5	0.89	0	0	-
11:00	1285	0	0.90	4.5	0.89	0	0	-
11:30	1415	0	0.90	4.5	0.91	0	0	-
12:00	1435	0	0.90	4.5	0.90	0	0	-
12:30	1485	0	0.90	4.5	0.90	0	0	-
12:45	1500	0.031	0.90	4.5	-	0	0	-
1:00	1515	0.031	0.90	4.5	0.92	0.4	0.01	12
1:15	1520	0.031	0.90	4.5	0.95	0.8	0.03	26
1:30	1529	0.031	0.90	4.5	0.95	1.7	0.08	56
1:45	1540	0.031	0.90	4.5	0.96	1.7	0.14	56
2:00	1545	0.031	0.90	4.5	0.95	1.6	0.20	53
2:15	1575	0.031	0.90	4.5	0.96	1.4	0.25	46
2:30	1610	0.031	0.90	4.5	0.96	1.3	0.29	44
2:45	1625	0.031	0.90	4.5	0.96	1.1	0.33	36
3:00	1630	0.031	0.90	4.5	0.95	1.5	0.38	50
3:15	1665	0.031	0.90	4.5	0.95	1.5	0.43	50
3:30	1695	0.031	0.90	4.5	0.90	1.2	0.47	38
3:45	1695	0	0.90	4.5	0.90	1.4	0.52	45
4:00	1710	0	0.90	4.5	0.90	0.7	0.54	21
4:15	1705	0	0.90	4.5	0.89	0.6	0.56	-
4:30	1705	0	0.90	4.5	0.89	0.5	0.58	-
4:45	1700	0	0.90	4.5	0.89	0.6	0.60	-

*Inlet Tube - same as for Run 29
Crucible - same as for Run 29
Charge - 70.0 g granite (no carbon)
CO_2 recovered = 0.10 g; H_2O recovered = 0.95 g.

TABLE 20

ROCK REACTOR DATA FOR RUN 31*

Time	Crucible Temp (°C)	Gas Flow Rates (scfh)				Carbon Monoxide Content in Product Gas (mole %)	Carbon Recovered as Carbon Monoxide (g C)	Methane Converted to Carbon Monoxide (mole %)
		Methane Feed	Argon Purge	H_2 Cooling	Product Gas			
11:30	900	0	0.90	3.4	0.92	0	0	-
12:00	1070	0	0.90	3.4	0.93	0	0	-
12:30	1223	0	0.90	3.4	0.92	0	0	-
1:00	1330	0	0.90	3.4	0.93	0	0	-
1:30	1440	0	0.90	3.4	0.92	0	0	-
2:00	1555	0.030	0.90	4.5	0.91	0	0	-
2:15	1570	0.030	0.90	4.5	0.92	0	0	-
2:30	1568	0.030	0.90	4.5	0.95	1.1	0.04	40
2:45	1545	0.030	0.90	4.5	0.94	1.8	0.10	62
3:00	1570	0.030	0.90	4.5	0.94	1.8	0.16	62
3:15	1585	0.045	0.90	4.5	0.98	2.2	0.24	79
3:30	1590	0.045	0.90	4.5	1.02	2.7	0.34	67
3:45	1585	0.045	0.90	4.5	0.98	2.2	0.41	52
4:00	1585	0.045	0.90	4.5	0.98	2.0	0.48	49
4:15	1590	0.060	0.90	6.0	1.01	1.9	0.55	46
4:30	1600	0.060	0.90	6.0	1.01	2.2	0.63	40
4:45	1615	0.060	0.90	6.0	1.01	2.4	0.74	45
5:00	1610	0.060	0.90	6.0	1.01	2.4	0.83	45

*Inlet Tube - same as Run 29
Crucible - same as Run 29
Charge - 70.0 g Indochinite tektites (no carbon)
CO_2 recovered = 0.16 g; H_2O recovered = 0.75 g.

TABLE 21

SPECTROGRAPHIC ANALYSES OF METAL OBTAINED FROM ROCK REACTOR MELTS

Run No.	Rock	Metal Composition (wt%)				
		Al	Fe	Si	Cu	Ni
31	Tektite	Not detected	92.7	5.0	0.2	2.0
32	Basalt	0.9	94.2	2.8	0.2	1.9

TABLE 22

ROCK REACTOR DATA FOR RUN 32*

Time	Crucible Temp (°C)	Gas Flow Rates (scfh)				Carbon Monoxide Content in Product Gas (mole %)	Carbon Recovered as Carbon Monoxide (g C)	Methane Converted to Carbon Monoxide (mole %)
		Methane Feed	Argon Purge	H_2 Cooling	Product Gas			
11:00	825	0	0.9	4.0	0.93	0	0	-
11:30	985	0	0.9	4.0	0.92	0	0	-
12:00	1085	0	0.9	4.0	0.91	0	0	-
12:30	1180	0	0.9	4.0	0.92	0	0	-
1:00	1340	0	0.9	4.0	0.91	0	0	-
1:30	1470	0	0.9	4.0	0.91	0	0	-
2:00	1555	0.027	0.9	6.0	0.90	0.1	0	-
2:30	1540	0.027	0.9	6.0	0.94	0.4	0.02	15
3:00	1565	0.027	0.9	6.0	0.94	1.9	0.14	72
3:30	1570	0.027	0.9	6.0	0.94	1.9	0.27	73
4:00	1585	0.027	0.9	6.0	0.94	2.0	0.41	73
4:30	1585	0.043	0.9	6.0	0.97	2.5	0.58	61
5:00	1585	0.043	0.9	6.0	0.97	2.6	0.76	63
5:30	1585	0.043	0.9	6.0	0.97	2.6	0.94	62
6:00	1605	0.043	0.9	6.0	0.97	2.4	1.11	58
6:30	1600	0.043	0.9	6.0	0.98	2.2	1.27	52
7:00	1570	0.043	0.9	6.0	0.99	1.9	1.41	47
7:30	1600	0.043	0.9	6.0	0.97	1.9	1.54	46
8:00	1590	0.043	0.9	6.0	0.97	1.9	1.68	45
8:30	1600	0.043	0.9	6.0	0.97	1.8	1.80	44
9:00	1605	0.043	0.9	6.0	0.98	2.0	1.94	48
9:30	1600	0.043	0.9	6.0	0.98	1.9	2.07	44

*Inlet Tube - triple-walled alumina terminating in a 1-in.-OD by 6-in.-long impervious ZrO_2 "bell."
Crucible - 1-1/2-in. + 3-in.-deep impervious ZrO_2
Charge - 70.00 g basalt (no carbon)
CO_2 recovered = 0.21 g; H_2O recovered = 1.43 g.

TABLE 23

ROCK REACTOR CARBON BALANCES BASED ON METHANE CHARGED

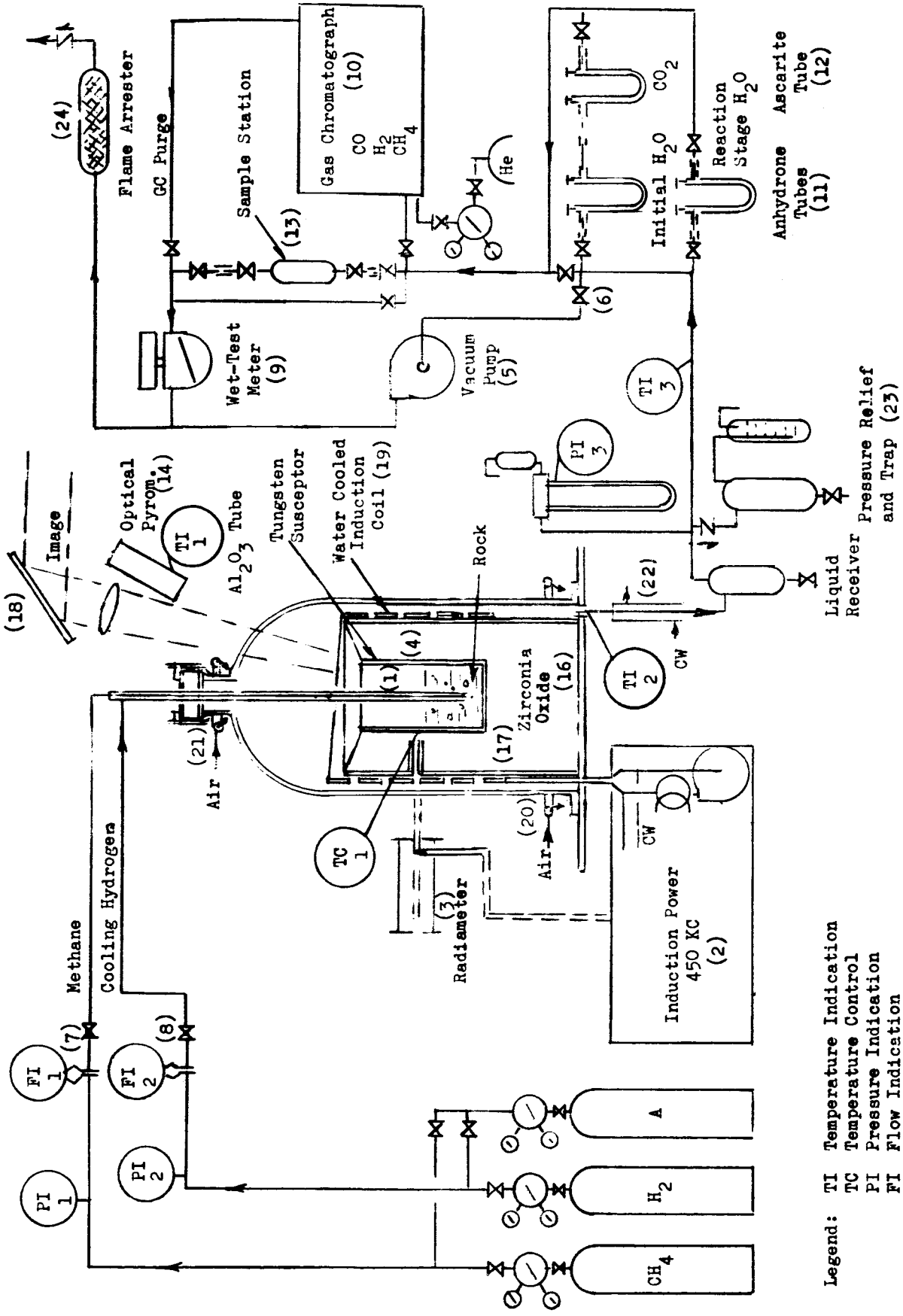
Run No.	Rock Charged Type	Rock Charged (g)	Carbon Charged as Methane (g C)	Carbon Recovered: As CO (g C)	As CO_2 (g C)	C as CO + CO_2 (%)	C in Outlet of Crucible (g C)	C in Inlet to Crucible (g C)	Total Carbon Recovered (g)	Total Carbon Recovered (%)
30	Granite	70.0	1.22	0.60	0.03	52	*	*	-	-
31	Tektites	70.0	1.63	0.83	0.04	53	0.04	0.71	1.62	99
32	Basalt	70.0	4.03	2.13	0.06	54	0.09	1.71	3.99	99

*Not determined.

TABLE 24

WATER RECOVERY DATA

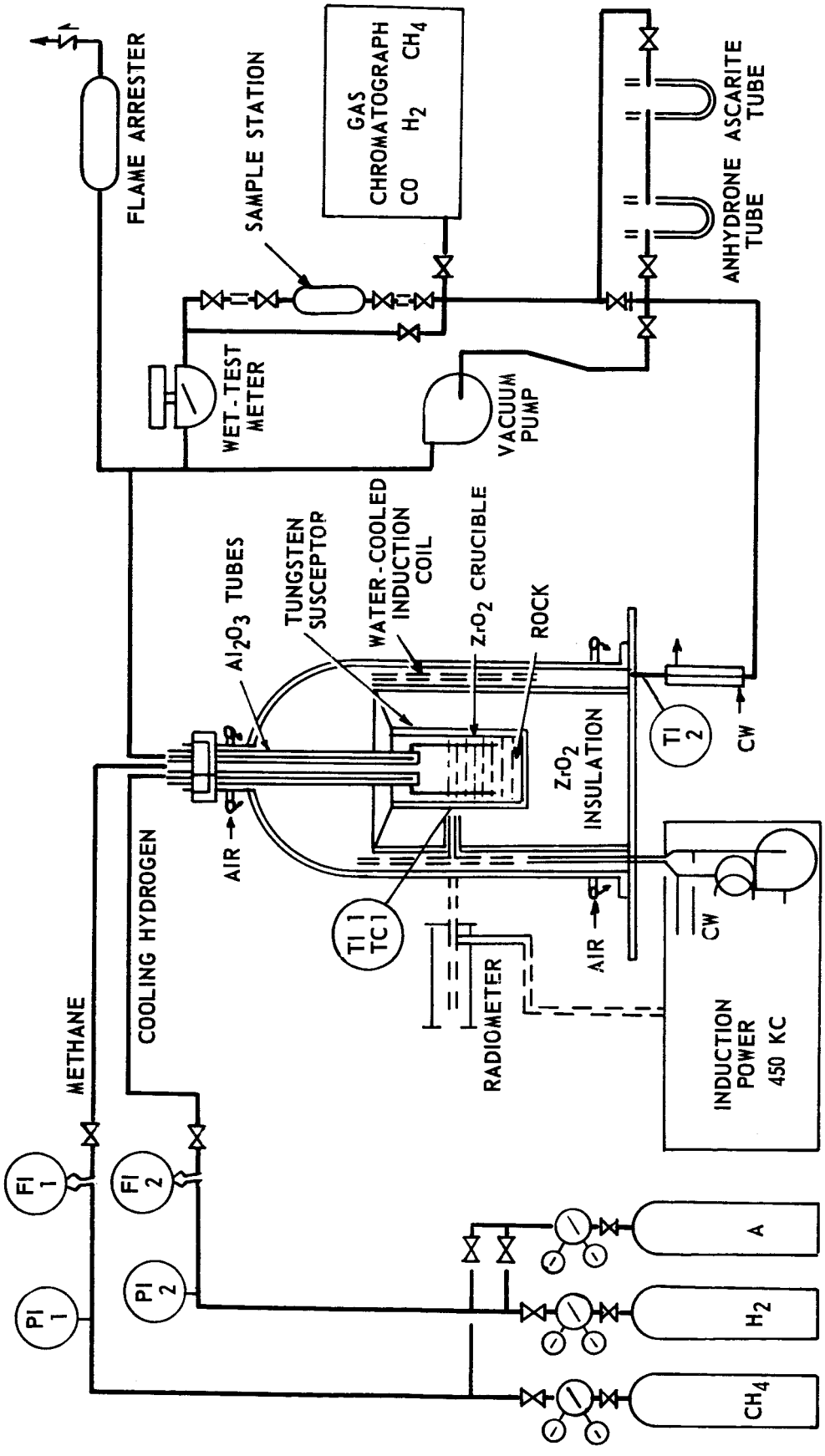
Run No.	Rock Charged Type	Rock Charged (g)	Form of Carbon	Water Recovered (g)
15	Basalt	99.1	Carbon + methane	1.91
16	Basalt	50.1	Carbon	0.43
17	Basalt	50.1	Carbon	0.08
18	Granite	50.0	Carbon	0.66
19	Basalt	50.0	Carbon	1.12
20	Granite	42.0	Carbon	1.39
22	Granite	37.5	Carbon	1.09
29	Basalt	69.9	Carbon + methane	1.15
30	Granite	70.0	Methane	0.95
31	Tektite	70.0	Methane	0.75
32	Tektite	70.0	Methane	1.43



Legend: TI Temperature Indication
TC Temperature Control
PI Pressure Indication
FI Flow Indication

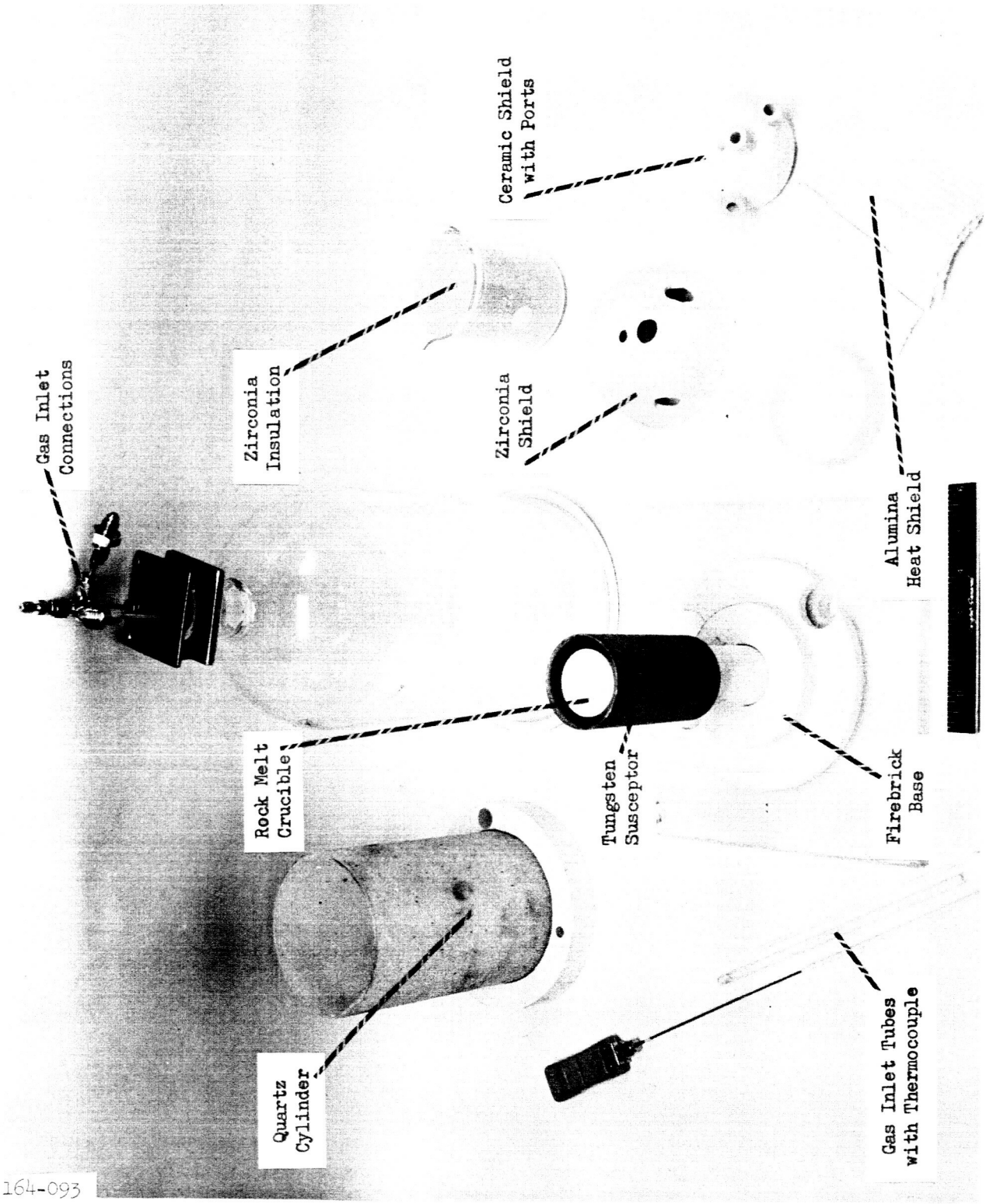
SCHEMATIC FLOW DIAGRAM OF SILICATE REDUCTION FURNACE
STEP 1, AEROJET CARBOTHERMAL PROCESS

Figure 1



Schematic Flow Diagram of Silicate Reduction Furnace

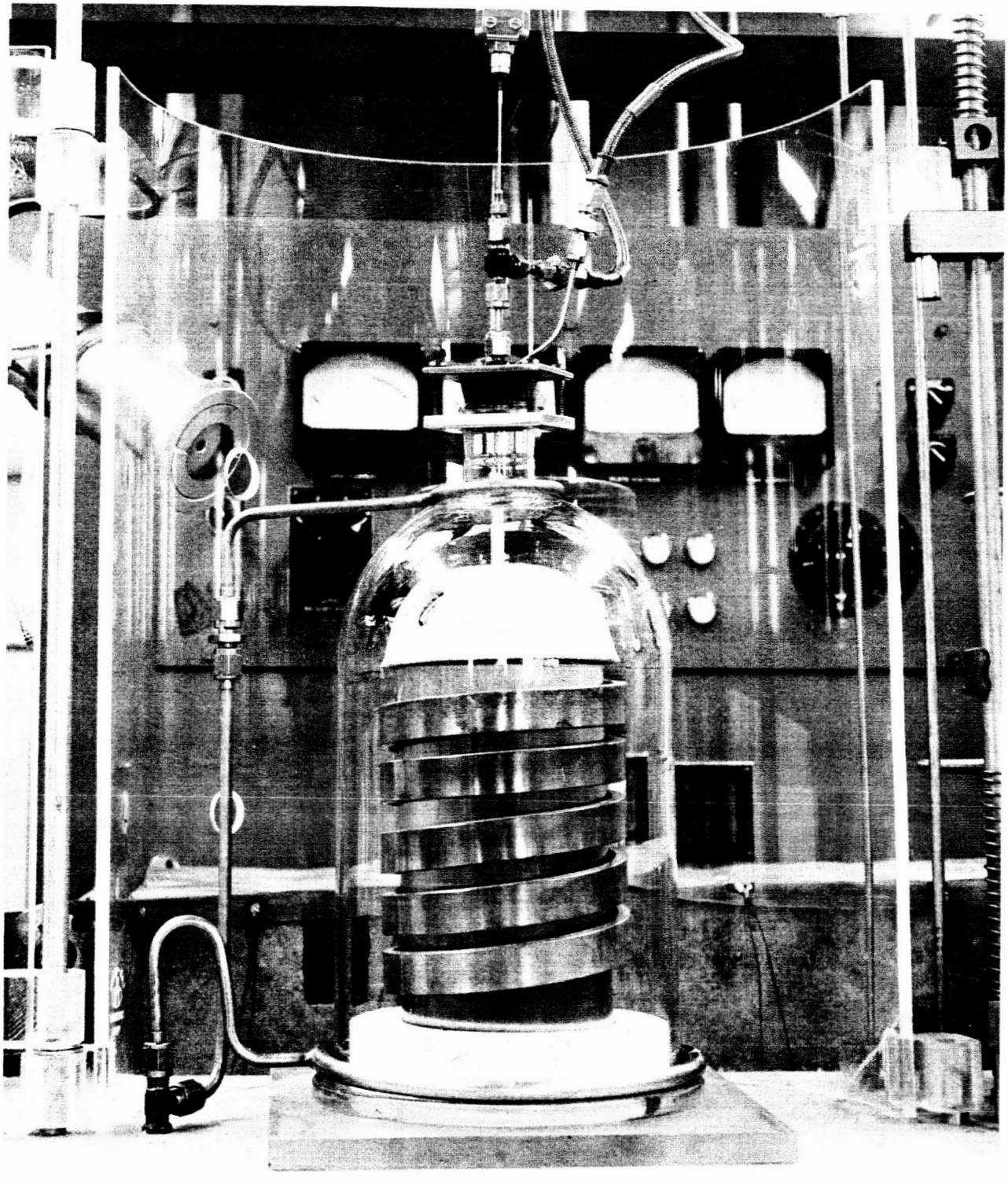
Figure 2



Reactor Parts

Silicate Rock Reduction Furnace

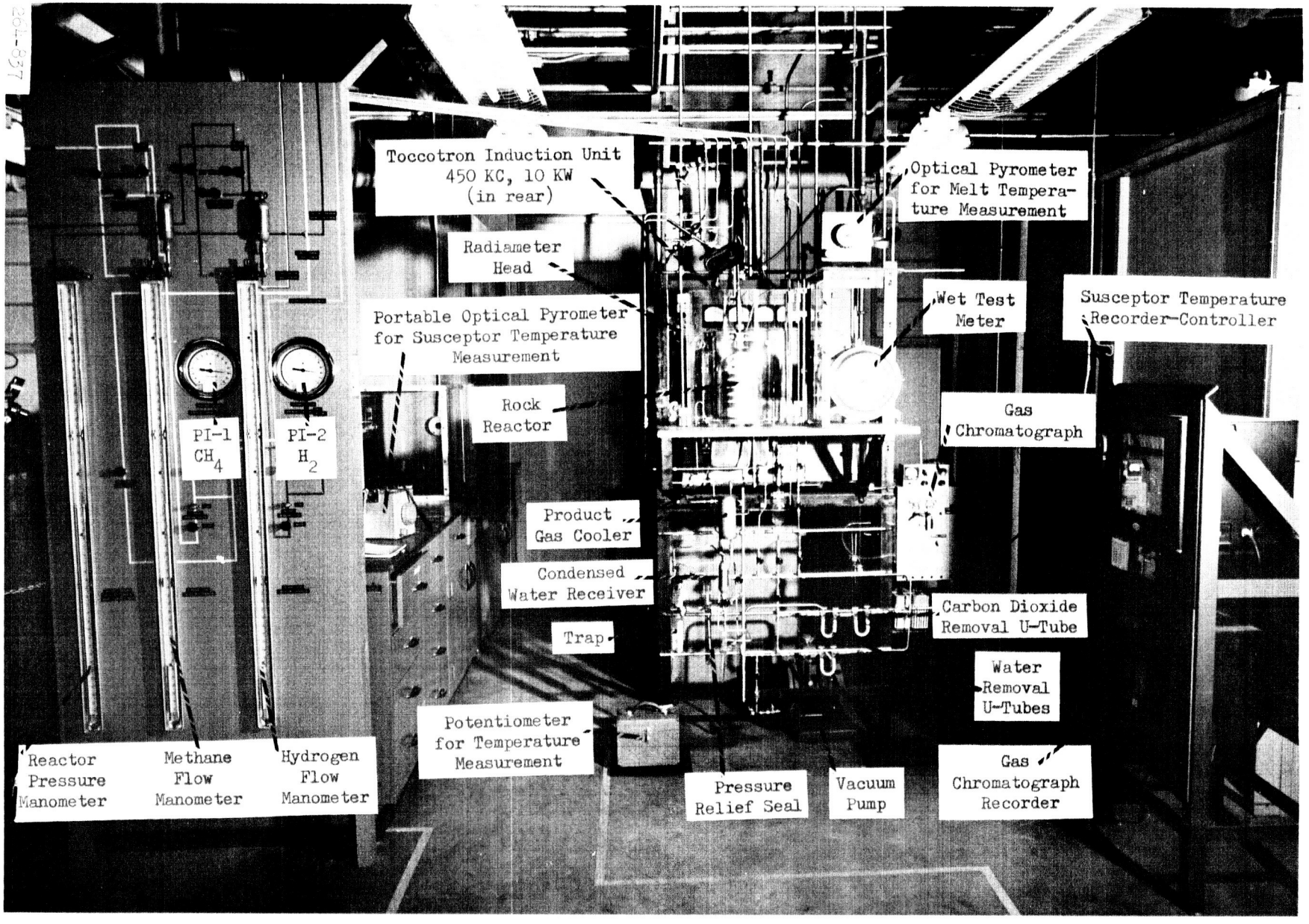
Figure 3



164-096

Assembled Rock Reduction Reactor, Silicate Rock Reduction Furnace

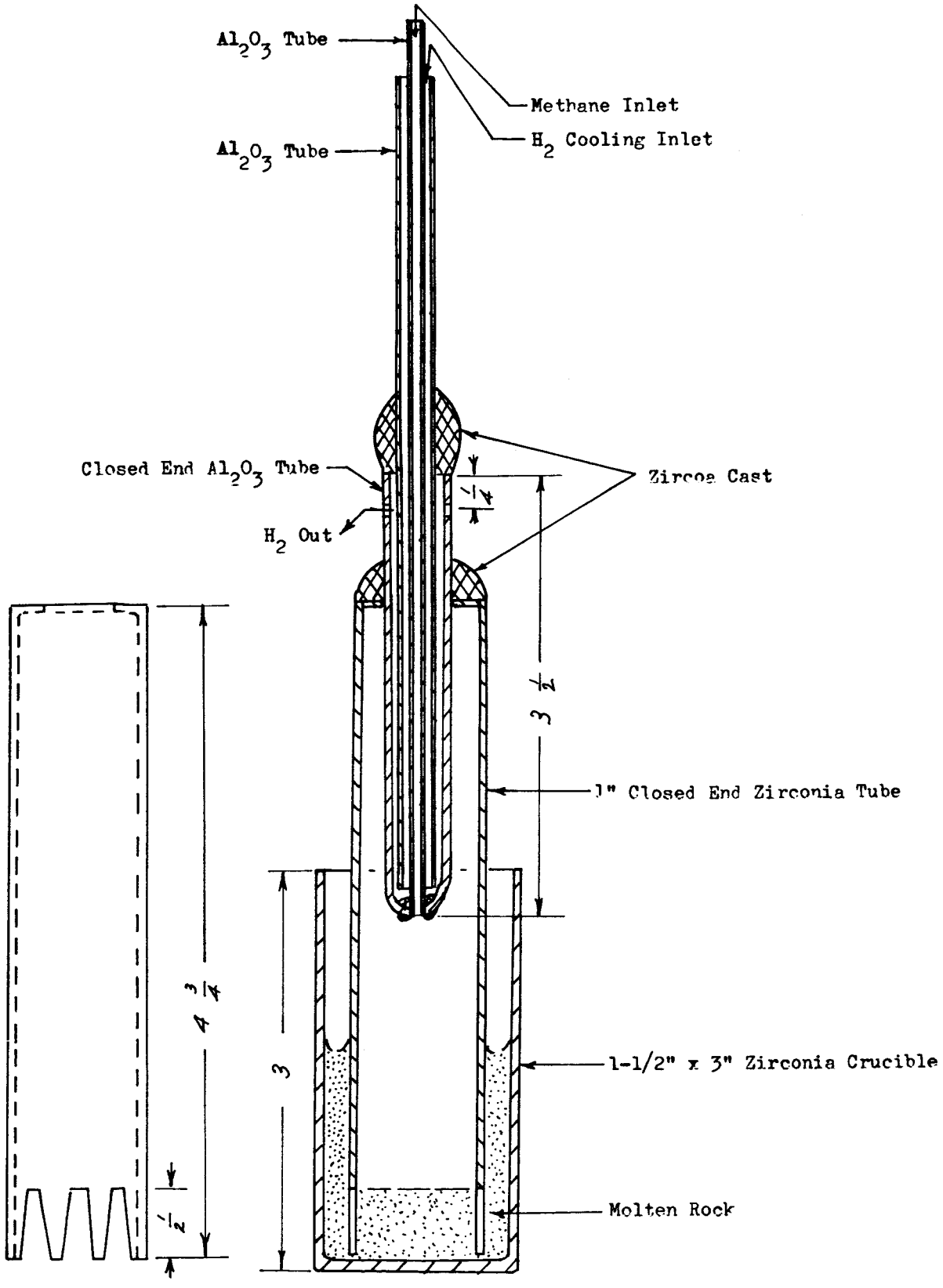
Figure 4



Silicate Rock Reduction Furnace

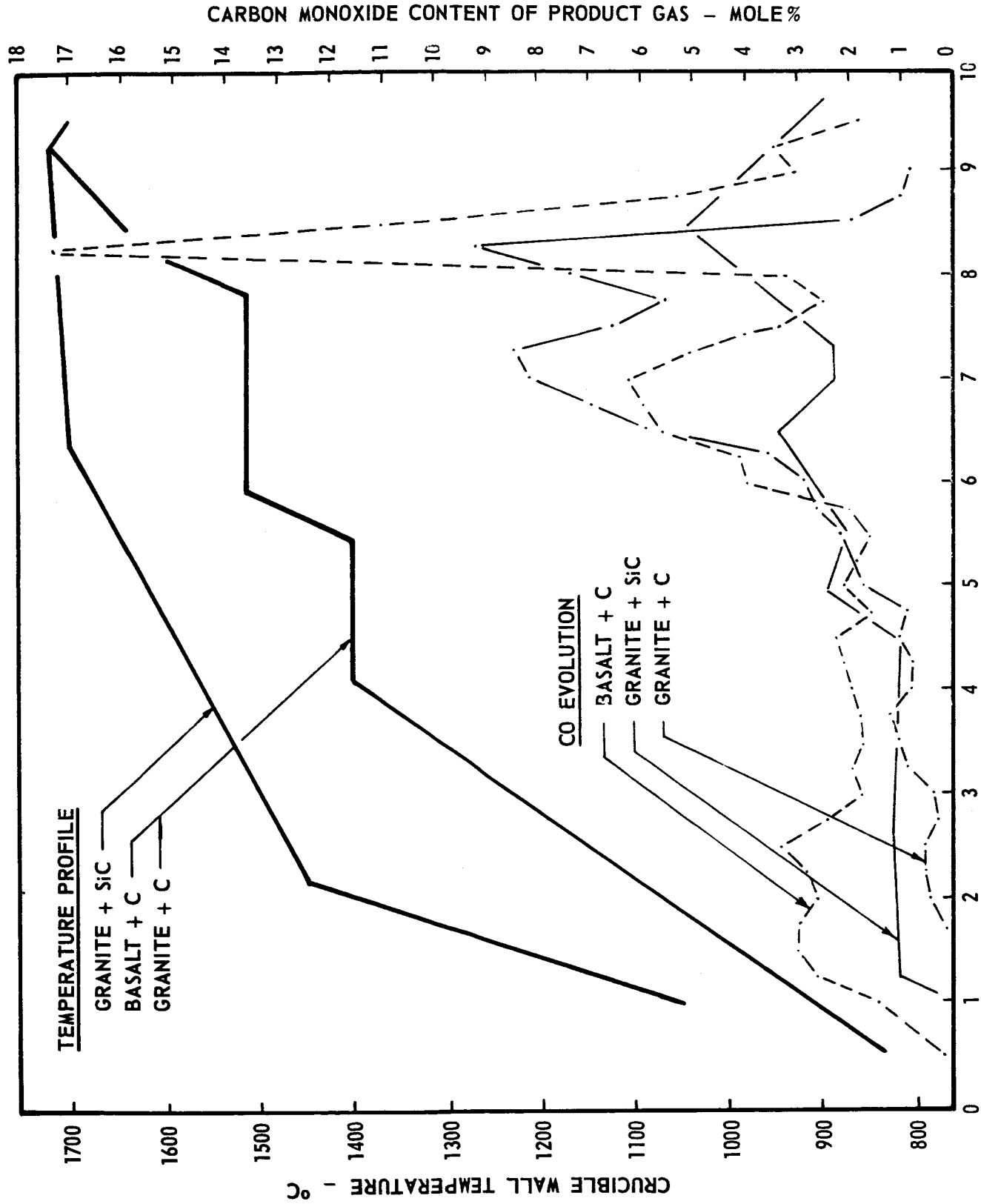
Figure 5

Report No. 2895



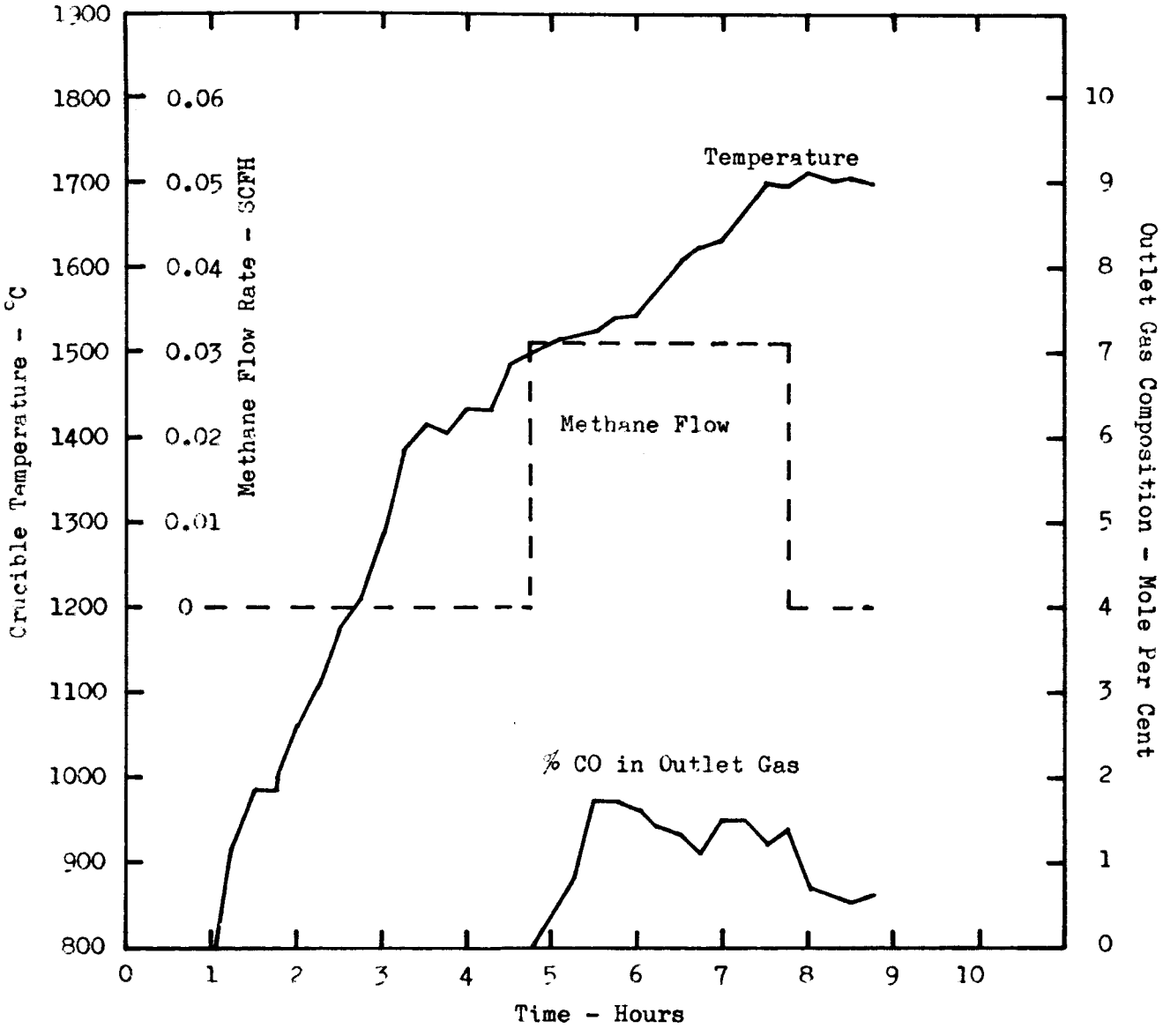
TRIPLE WALLED INLET TUBE WITH "BELL"

Figure 6



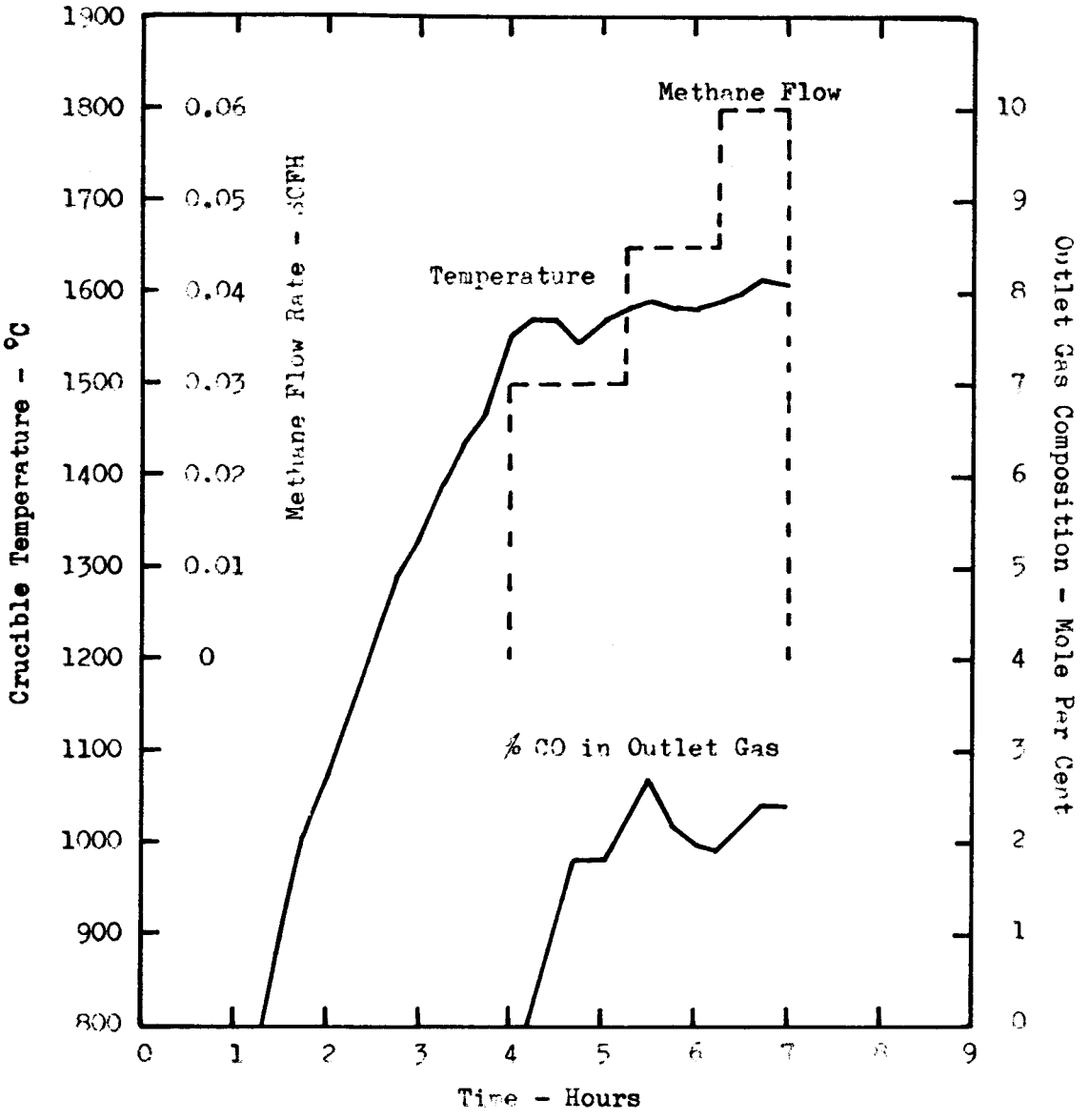
Reduction of Igneous Rock with Carbon and Silicon Carbide

Figure 7



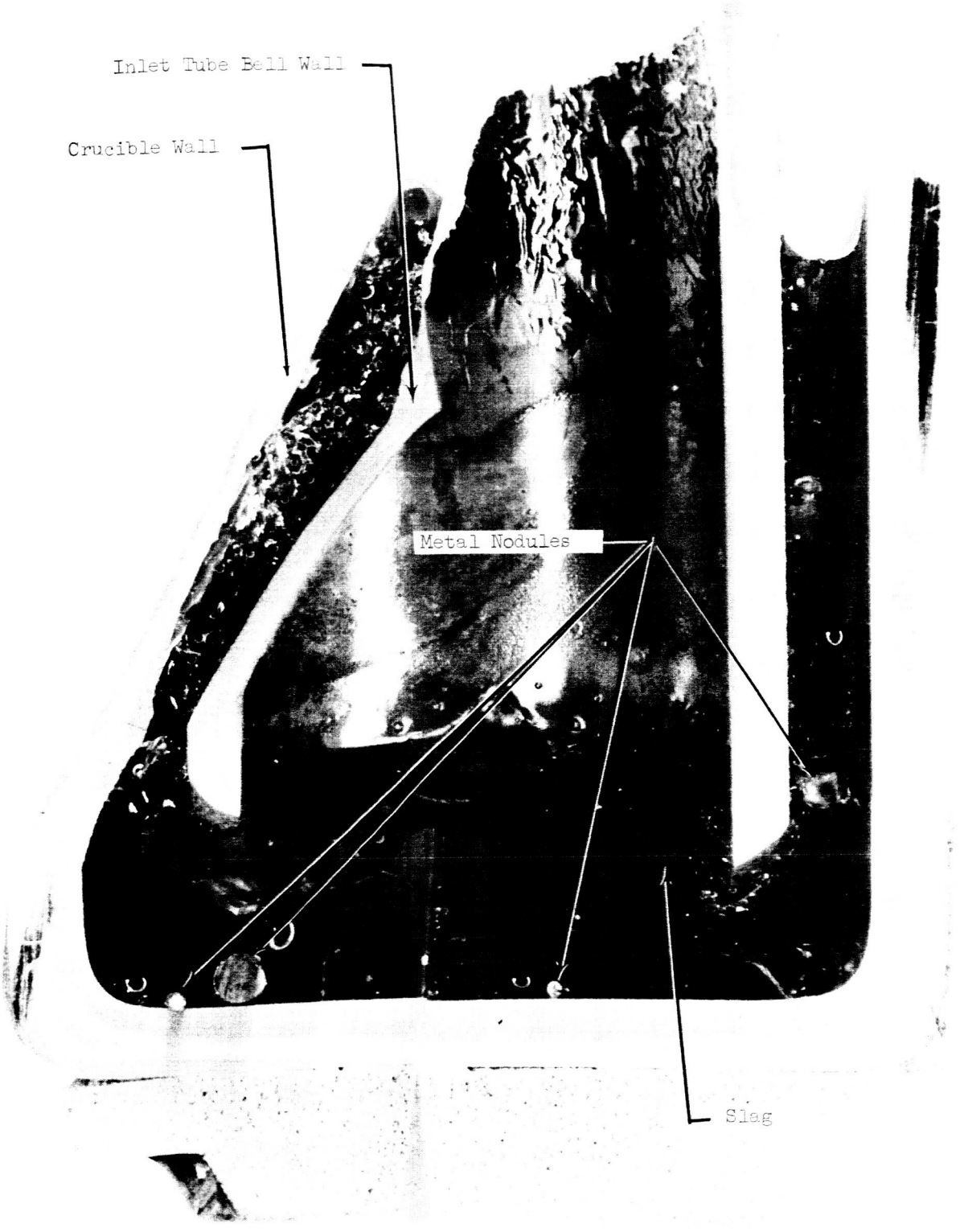
LOG OF RUN 30 - REDUCTION OF GRANITE WITH METHANE

Figure 8



LOG OF RUN 31 - REDUCTION OF TEKTITES WITH METHANE

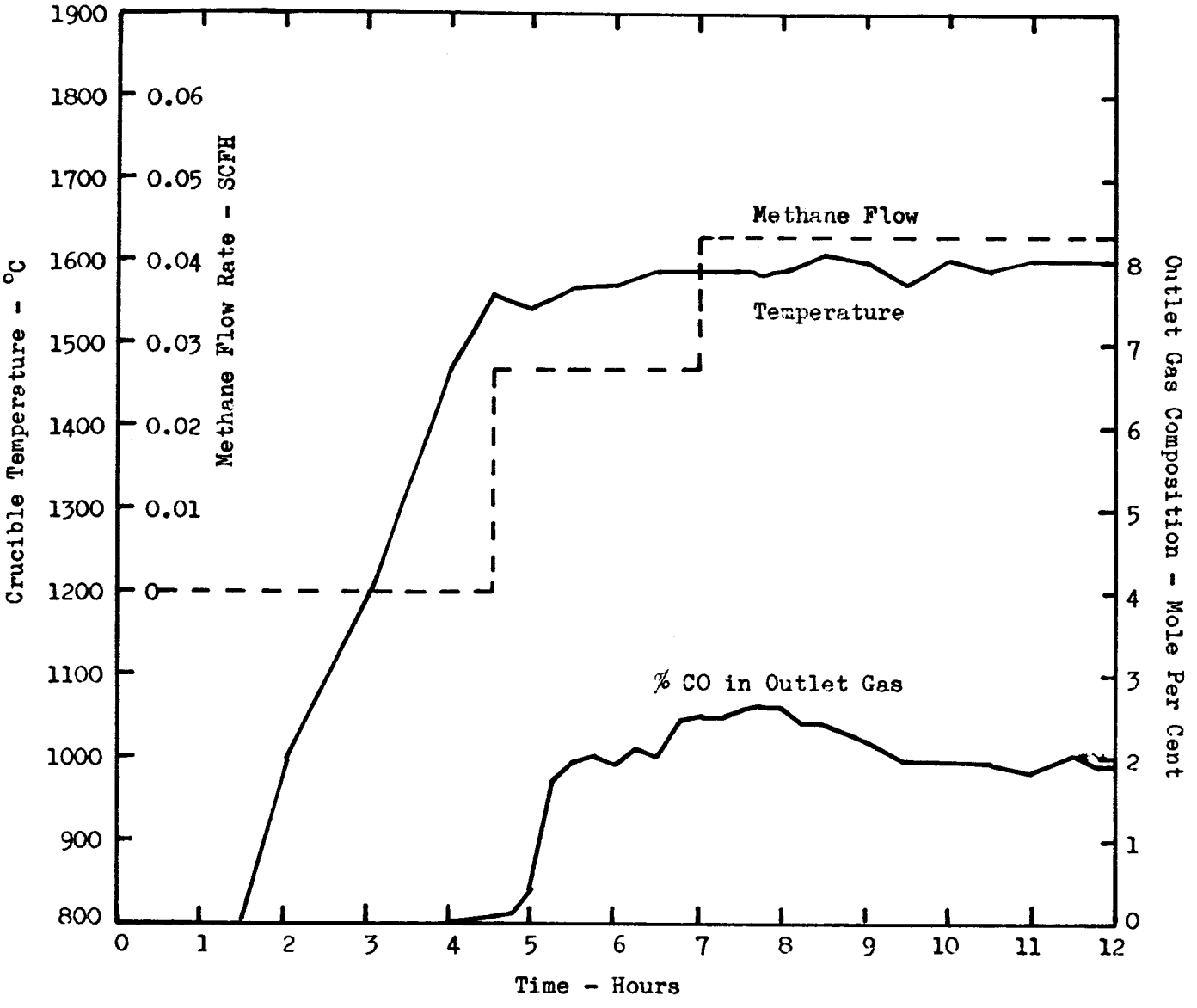
Figure 9



Nodule Analysis: 92.7% Fe
5.0% Si
2.0% Ni
0.2% Cu

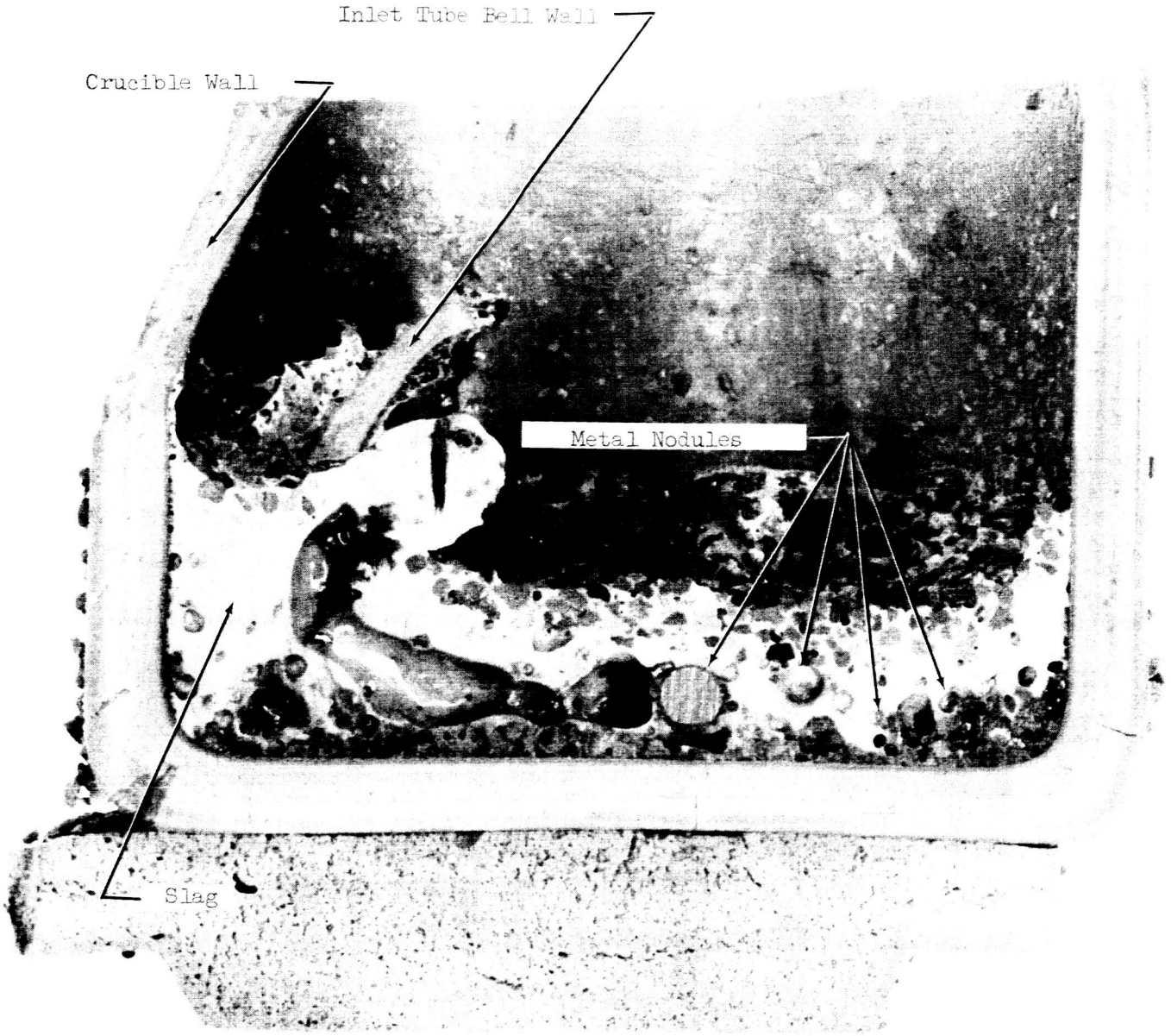
Crucible Section Containing Tektite Melt After Reduction with Methane (Run 31)

Figure 10



LOG OF RUN 32 - REDUCTION OF BASALT WITH METHANE

Figure 11



Nodule Analysis:
94.2% Fe
2.8% Si
1.9% Ni
0.9% Al
0.2% Cu

Crucible Section Containing Basalt Melt After Reduction with Methane (Run 32)

Figure 12

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